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Sediment Sampling Guide for Dredging and Marine Engineering Projects in the St. Lawrence River

Volume 2: Field Operations manual

Canada

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Sediment Sampling Guide for Dredging and Marine Engineering Projects in the St. Lawrence River

Volume 2: Field Operations manual

Environmental Protection Branch
Environment Canada, Quebec Region
Montreal, Quebec

October 2002

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Correct citation for this publication:
Environment Canada. 2002. *Sediment Sampling Guide for Dredging and Marine Engineering Projects in the St. Lawrence River. Volume 2: Field Operations Manual*. Environment Canada, Environmental Protection Branch, Quebec Region, Technological Innovation and Industrial Sectors Section. Report 103 pages.

Published by authorization of the Minister of the Environment
© Minister of Public Works and Government Services Canada
Catalogue No.: En154-1/2002-2E
ISBN: 0-662-33062-5

Acknowledgments

This guide was drafted from a synthesis prepared by Stéphane Lorrain, of the firm Service d'Études Sédimentologiques, a division of Environnement Illimité Inc. of Montreal, Quebec. We would like to thank Mr. Lorrain whose expertise and quality of work played an important role in the elaboration of this document.

Michel Chevalier and Julie Leduc of Environment Canada's Environmental Protection Branch oversaw the scientific aspects of the work. Throughout the project, technical advice was provided by an ad hoc project monitoring team composed of Serge Morissette and Mireille Blouin of the Centre d'Expertise en Analyse Environnementale du Québec, Yves Lavergne of Public Works and Government Services Canada, and Suzie Thibodeau of Environment Canada's Environmental Protection Branch.

We would like to thank Serge Morissette for his extensive contributions in statistics, particularly for drafting Appendix C (volume 1), which deals with the cost of uncertainty in environmental sampling. We also thank Caroll Bélanger and Alain Latreille of Environment Canada's Environmental Protection Branch, Pierre Gagnon, Christian Gagnon, Isabelle Saulnier, Louis-Filip Richard and Jean-François Bibeault of Environment Canada's St. Lawrence Centre, Pierre Michon of the Quebec Environment Ministry, Yves Lavergne and Marc Desrosiers of Public Works and Government Services Canada, Pierre Rouleau of the Canadian Coast Guard, Marc Pelletier of Procéan, and Jacques Bérubé of Consultants JBL Inc. for their comments and suggestions. We are grateful to Monique Simond of Environment Canada's St. Lawrence Centre for editing and formatting the guide, Public Works and Government Services Canada, Translation Services, for the English translation, Patricia Potvin of Environment Canada's St. Lawrence Centre for editing the translated text and Dianne Ouelle and Martine Bluteau of Environment Canada's Environmental Protection Branch for the publication of this guide.

Lastly, we thank the members of the Working Group on the Integrated Management of Dredging and Sediments (a group formed to support the development of the Sustainable Navigation Strategy for the St. Lawrence) for the final editing of the guide and for encouraging its use on the St. Lawrence River.

Foreword

The purpose of this document is intended to help proponents and managers of dredging and marine engineering projects to design and implement sediment sampling plans that satisfy concerns about the physical and chemical characterization of sediments. The guidelines are accompanied by explanations and advice, to enhance the guide's efficiency.

This guide also complements the bioassay methods published by Environment Canada for toxicity and bioaccumulation assays on pore water and whole sediments. The standardized approaches and operating methods suggested here must be followed to ensure the standardization of procedures for sample collection and the documentation of characterization work. This will facilitate successful sampling and improve the acceptability of results.

The use of this guide for sampling sediments dredged from the St. Lawrence River is recommended by Environment Canada, the Quebec Environment Ministry, Fisheries and Oceans Canada, Transport Canada, Public Works and Government Services Canada, and the Société de la Faune et des Parcs du Québec (Quebec Wildlife and Parks Corporation).

Abstract

The approach used to characterize sediments in dredging and marine engineering projects has been revised and updated in a new two-volume methods manual. The first volume, *Planning Guidelines*, is intended for planners of characterization studies, while this volume, *Field Operations Manual*, is addressed to the technical teams carrying out the actual sampling work. The use of this guide is recommended to ensure the standardization of sample collection procedures and the documentation of sediment characterization work.

This volume deals mainly with field-related aspects of sampling and the preparation of sediment and pore water samples. It is divided into eight chapters. The introduction describes the relevance of the guide and the context in which it was written in order to define the requirements involved in carrying out characterization studies in a standardized framework. The next three chapters deal with conducting a sampling campaign and quality control and health and safety in the field. The fifth chapter describes sampling methods, including general field procedures and positioning methods, while the sixth deals with handling samples. The last two chapters address field observations and measurements and sample preservation methods.

Résumé

L'approche de caractérisation des sédiments dans le cadre des projets de dragage et de génie maritime a été revue et mise à jour dans un nouveau guide méthodologique présenté en deux volumes. Le premier volume, intitulé « *Directives de planification* », est destiné aux planificateurs de l'étude de caractérisation alors que le second, intitulé « *Manuel du praticien de terrain* », est destiné aux équipes techniques chargées des travaux d'échantillonnage. L'utilisation du guide méthodologique est recommandée pour assurer une normalisation des procédures de collecte des échantillons et de documentation des travaux de caractérisation des sédiments.

Le manuel de terrain, qui comprend huit (8) chapitres, vise essentiellement les aspects de l'échantillonnage et de la préparation des échantillons de sédiments et d'eau de porosité sur le terrain. Dans l'introduction, on présente l'à-propos de ce guide ainsi que le contexte dans lequel il a été élaboré afin de définir les contraintes entourant la réalisation d'études de caractérisation dans un cadre normalisé. Les trois chapitres suivants traitent de la campagne d'échantillonnage, du contrôle de la qualité sur le terrain et de la santé et sécurité sur le terrain. Le cinquième chapitre vise les méthodes d'échantillonnage, incluant les procédures générales de terrain et les méthodes de positionnement, et le sixième, la manipulation des échantillons. Les deux derniers chapitres ont trait aux mesures et observation de terrain ainsi qu'aux modes de conservation des échantillons.

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Glossary

Aliquot: Quantity of homogeneous material, taken for analysis purposes, with a supposedly negligible sampling error. Usually applies to fluids.

Artefact: Undesirable characteristic (e.g. chemical or physical modification) detectable in a substrate and caused by local phenomena or the handling of the substrate.

AVS: Acid volatile sulfide. Chemical form of trace metals present in pore water, in which the trace elements are bound to sulfur and therefore do not contribute to the overall toxicity of the sample.

CAB: Cellulose acetate butyrate. Transparent plastic material used to make the internal liners (liner tubes) of some corers.

CEC: Cation exchange capacity. Measurement of cation adsorption potential of sediments, particularly clay minerals with a high surface-to-volume ratio.

Cohesive sediments: Sediments demonstrating a certain degree of cohesion due to a bond between particles. Cohesive properties are usually observed in sediments with more than 15% clay.

Composite sample: Sample composed of sediments from different sites in equal proportions that is then homogenized to incorporate intrasite variability.

Conspicuous beacon: Landmarks on the shoreline or surface of the water allowing the position of sampling stations to be precisely determined.

Contaminant: Undesirable agent, substance or material present in sediments or water.

Corer: Type of sampler used to collect a core sample to describe sediment properties in a given thickness of the sediment column.

Cross contamination: Cross contamination occurs when a sample from an uncontaminated or relatively uncontaminated station comes in contact with a sample from a more contaminated station. The correct cleaning of the various sampling instruments reduces the risk of cross contamination.

Dummy sample: Sample identified so that the laboratory cannot determine the origin. Accompanying a sample from the same site, it is used in the analytic quality control program to verify the reproducibility of the results.

Ecotoxicological assay: Series of tests used to assess the degree of toxicity of sediments or pore water using a biological substrate (bacteria, more or less highly evolved organisms, etc.)

Eh (oxidation-reduction potential or redox potential): Measurement in volts of the value of the oxidizing power of a medium.

Electro-osmotic spatula: Spatula used in core subsampling. A weak electric current between the spatula and a rod inserted in the core is used to facilitate the division of the core into two parts.

Elutriate: Aqueous solution obtained by adding water to a solid or unconsolidated material (e.g. sediments, drilling mud, dredging materials), stirring the mixture and then by centrifuging, filtering or decanting the supernatant.

Field blank: Sample of distilled deionized water that is exposed in the field during all stages of sampling to detect possible contamination during the operations. The field blank may also be made up of rinse water from the sampler to detect potential contamination in it.

Glove box: Leak-tight container with a controlled atmosphere, the inside of which can be accessed through gloves tightly sealed around openings in the wall of the box.

GPS (Global Positioning System): Positioning system using satellites. Positions are obtained instantaneously from a constellation of satellites. Position accuracy and reproducibility can be improved by using differential correction.

Grab sampler: Sampler with a set of jaws, which can be used to collect samples of surface sediments, generally in large volumes.

HDPE (High-density polyethylene): Relatively inert plastic material used to manufacture subsampling containers and tools.

Lexan: Plastic material made of polycarbonate.

Physicochemical characterization: Series of physical and chemical analyses used to identify sediment properties.

Pore water *(*syn.: *interstitial water)*: Water occupying the space between sediment particles. It can be directly extracted in the field or the laboratory.

PTFE: Plastic material made of polytetrafluoroethylene. It is a relatively inert substance with properties similar to Teflon®.

Reference site: Sediment sampling site used to provide sediments with the same physicochemical properties as the sediments in the site under characterization, except for the presence of contaminants. The reference sediments are used to validate bioassay results.

Replicate sample: In sediment sampling, used to refer to each reserve or supplementary sample taken at the same sampling station for analytic quality control purposes. It may be a field replicate, obtained from the repeated independent sampling of the same station, or a split sample (pseudo-replicate), obtained by dividing the same sample into at least two distinct parts (subsamples).

Sample: Representative part of a larger whole (e.g. sediments, pore water) that is studied to better understand the characteristics, and infer the properties, of the whole. Also refers to a subset of a population (e.g. benthos).

Sampling plan stratum: Subzone in a sampling plan in which the characteristics of the environment and/or sediments are homogeneous and different from adjacent strata. A stratified sampling plan is used to optimize the distribution of stations on the sampling grid to maximize the informational value of the samples.

Sampling station: Site for taking samples in the study area.

Spot sample: Sample obtained from a single sampling station (it may also be obtained from several different passes of the same sampler at the same station).

Standard operating procedure (SOP): Protocol detailing the steps in an activity (e.g. sediment sampling using a given sampler). This protocol is used to ensure that, if these procedures are followed, all sampling activities will be carried out in an identical fashion at all stations and the information obtained will be homogeneous.

Subsample: Representative part of a sample that is studied to better understand the characteristics, and infer the properties, of the whole.

Teflon®: Plastic material derived from fluorine and ethylene and highly resistant to chemical agents.

TET: Toxic effect threshold. Third level of toxicity for evaluating sediment quality in the St. Lawrence River under the interim criteria.

1 Introduction

Characterization studies are intended to respond to the concerns of stakeholders regarding the protection of the integrity and health of aquatic ecosystems. To this end, different sediment and pore water sampling methods can be used to carry out physicochemical characterizations, ecotoxicological assessments or bioaccumulation measurements. The activities that these studies entail, however, are likely to influence the physical and chemical properties of sediments and, consequently, the results of the analyses. The purpose of developing standard operating procedures (SOPs), therefore, is to allow comparable results to be produced even from different sediment sampling campaigns.

The objective of the *Sediment Sampling Guide for Dredging and Marine Engineering Projects in the St. Lawrence River* is to provide guidelines to ensure that sampling and sampling handling methods are standardized, reproducible and effective, and carried out within the framework of a quality assurance and control program.

The technical information presented in this document generally follows the steps involved in an actual sampling campaign carried out in the field. On the other hand, sampling activities must be viewed in the larger framework of project planning, which in the case of a characterization study includes such aspects as analysis, the processing and interpreting of the results and the development of methods for managing potentially contaminated sediments.

2 Sampling Campaign

2.1 OBJECTIVES

Since the primary objective of any sampling campaign is to maximize the informational value of the samples, it is crucial that the latter be representative in order to ensure the accurate interpretation of the results. Therefore, great care should be taken in describing the sample characteristics and sampling conditions and in evaluating sampler performance, to ensure that the data quality objectives for the project are met. The technical methods and guidelines presented here, which involve various aspects of a sampling campaign, are currently considered to be the most appropriate for dredging and marine engineering projects in the St. Lawrence River.

2.2 SAMPLING PLAN

The number and location of the sediment sampling stations are defined in the specific sampling plan for the project. This plan is formulated based on the project objectives and other factors described in detail in volume I (Sampling Guidelines) of the *Sediment Sampling Guide for Dredging and Marine Engineering Projects in the St. Lawrence River.* Although the plan should be implemented systematically, a certain degree of flexibility must be allowed in order to adapt the sampling approach to the environmental conditions present.

2.3 TYPES OF SAMPLES

Depending on the study design objectives, two types of samples can be collected at a given station:

- spot sample
- composite sample.

A *spot sample* comes from a single sampling station but may be obtained with one or more passes of the sampler. In the latter case, the boat may be moved a small distance to ensure that successive passes do not collect material reworked by previous passes. A *composite sample* differs from the above in that it is composed of sediments from different sites in equal proportions. The sediments are then homogenized to incorporate intrasite

variability. The sediments can come from a single station of a given area, or from different stations that are to be integrated into a single stratum of the sampling plan. In the latter case, the boat must be moved from station to station and the positions of the points where sediments are collected must be recorded.

2.4 SAMPLING EFFORT

When sampling at a station, taking a supplementary or reserve sample can be useful for quality control purposes. Such analytical replicates are of two types:

- a *field replicate*, obtained from a reserve or supplementary sample and which results from the independent sampling of the same station
- a *split sample* (pseudo-replicate), obtained by dividing the same sample into at least two distinct parts (subsamples).

In the first case, variations in the results will be due to the techniques used for collecting and dividing the samples, as well as the heterogeneity of sediments within a single sampling station.

Split samples are used to determine the variance resulting from homogenization and analysis. This type of replicate cannot be used to determine intrasite variations in sediment properties.

To determine the level of homogeneity (or heterogeneity) of sediment properties within a given station or stratum (intrasite and intersite variability), the results from replicates—in other words, samples that undergo the same stages of sampling and subsampling at a given station—must be compared. By determining the intrasite and intersite variability, the user can define a concentration variation interval for each stratum and for the study area as a whole. Replicate samples should be taken from at least one station per stratum. At least three replicates and preferably five should be taken and each should be homogenized separately. The vessel should be moved a small distance between each replicate to avoid sampling sediments disturbed by the previous sampling operation.

When contaminant levels exceed Minimal Effect Thresholds (MET), additional sampling must be carried out for ecotoxicological assays. These assays involve different sample collection and handling requirements, including such things as the type of sampler used, volume of samples required, handling and subsampling procedures and the measurements and observations required during sampling. Taking samples at one or more

reference sites is also required. The environmental characteristics of these sites should be as similar as possible to those in the dredging area. The location of these sites must be verified in the field, usually according to the description of the characteristics of the samples collected. In general, sites are preferred that are near the study area but relatively free from the influence of sources of contamination. Reference sites should always be sampled before stations in the dredging area to limit the risks of cross contamination.

2.5 ANALYSIS PARAMETERS

Determining the parameters to be analysed is a critical part of planning a sampling campaign. Given the high cost of chemical analyses, judicious choices are required. Tables E.1, E.2 and E.4 (Appendix E) show the main analytical parameters for sediments and pore water. Documents such as *Interim Criteria for Quality Assessment of St. Lawrence River Sediments* (1992) and *Methods Manual for Sediment Characterization* (1993) also provide suggestions on the parameters to be analysed and analysis methods to be used.

3 Quality Control in the Field

The scope of the quality control program depends on the scope of the sampling program itself, as well as requirements dictated by the data quality objectives. This section describes commonly used quality control samples such as blanks (transport, field and equipment blanks) and field duplicates. Depending on the scale of the sampling campaign, additional control elements used to pursue specific objectives can also be added to assist in the interpretation of results.

A *transport blank* is used to determine if any contamination of containers or samples has occurred during transport. It is a liquid (water) or solid (uncontaminated sand or sediments) sample that accompanies regular samples during transport.

A *field blank* is used to verify if contamination has occurred during sampling. The container of the blank is opened in the field during sampling and homogenization.

An *equipment (rinsate) blank* is used to evaluate the effectiveness of procedures for cleaning equipment. When required, it is taken at the beginning of the sampling campaign, during personnel changes or when there is doubt over equipment contamination.

The contamination of any one of these blanks may indicate that all samples are contaminated.

A *field duplicate* (or triplicate, etc.) is a reserve or supplementary sample used for analytic quality control purposes. It is used to establish the replicability (if analysed at the same laboratory) or reproducibility (if analysed at two different laboratories) of the sampling work. Replicates should be as representative as possible of the original sample and duplicate samples should be sent to the laboratory under two different identification codes. As contamination is rarely homogeneously distributed among solids, however, duplicate sampling in the field may lead to the obtainment of very different results and unreliable conclusions. In the case of solids, then, it is preferable to define a field duplicate as two subsamples taken from a single homogenized (spot or composite) sample.

4 Health and Safety in the Field

Since health and safety procedures in the field are beyond the purview of this guide, only a brief description will be given, focusing on sediment contamination. For more information, consult Environment Canada's *Inspector's Safety Guide* (1995).

The field team should have the experience required to assess working conditions and be familiar with the operation of the boats, hoisting devices and samplers used in the project.

The development of standard operating procedures (SOPs) should include a health and safety component targeting the general aspects of work in an aquatic environment (water safety, personal safety and survival equipment, emergency procedures, communication system, navigational instruments, etc.) and certain aspects of overall field work (first aid, handling samplers, use of reagents). A list of emergency telephone numbers and contacts should also be drawn up.

The handling of potentially contaminated samples requires implementing certain health and safety rules:

- Anyone handling samples must wear safety gloves. To prevent sample contamination, gloves coated with powder (e.g. latex surgical gloves) are forbidden.
- When describing samples, special care should be taken in identifying odours since some sediments can generate strong odours or give off volatile substances.
- Since some contaminants can cause skin irritation, workers should wear safety goggles and protective clothing to provide protection in case sediments splatter.
- Handling chemical products (solvents, acids) in the field requires the proper identification of containers and bottles.
- Used chemical products must be disposed of in appropriate containers and correctly identified. An agreement with the laboratory should include the management of contaminated wastes and chemical products.
- The laboratory should be advised if high concentrations of contaminants in sediments are suspected.

Used acids and solvents must also be transported offsite and disposed of in accordance with current regulations, guidelines and municipal bylaws. Containers for these products must be stored according to safety standards. Emergency procedures in the event of a spill should also be established.

5 Sampling Methods

5.1 SEDIMENT SAMPLING

Two main types of samplers are suitable for sediment characterization studies carried out as part of a dredging project: grab samplers and corers. Their general characteristics and applications are described in sections 5.1.1 and 5.2.2. The choice of a sampler should be based on a number of factors:

- Hoisting equipment required
  The type of hoisting equipment required may be a significant constraint in terms of the type of vessel used, the experience of the field team in operating hoisting equipment and the resulting costs.
- Physical constraints
  Physical constraints that can limit the use of a sampler include depth, currents, waves and sediment characteristics.
- Penetration depth
  The penetration depth of some samplers can be controlled (e.g., vibro-corers, drilling) while, in others, depth is determined by the nature of sediments and degree of sediment consolidation.
- Recovery
  The sample recovery rate is determined by the quantity of materials present in the sampler. If the sampler is full, the recovery rate is 100%. Poor recovery indicates sampler malfunction, losses during recovery or simply that the sampler is not adapted to the type of sediments found.
- Sample integrity
  Sample integrity is an important aspect to be taken into account, particularly in the case of poor recovery. If the sampler is not completely closed, the washout of the fine fraction may occur when the sampler is being retrieved, introducing a bias in the sample.
- Composition of sampler
  The material of which the sampler is composed must also be considered to avoid any possibility of sample contamination. Sediments collected for an analysis of metals should not come into contact with metal materials, for example. Similarly, sediments taken for an analysis of organic substances should not come into contact with any plastics. However, some samplers may be used if they have a Teflon® lining or are made of stainless steel. Adequate quality control and subsampling techniques will also minimize contamination risks.
- Sample volume
  The volume required depends on the type and number of analyses.

The choice of sampler should also be based on the study objectives (surface sampling, dredging depth or both) and the information available on the nature of the

sediments to be removed. If sediment types vary, it may be necessary to use more than one type of sampler. The characteristics of an ideal sampler are shown in Table 5.1

Table 5.1
Ideal characteristics of a sediment sampler

- Allows free flow-through of water during descent, to avoid generating a shock wave
- Equipped with a straight-angle cutting edge, smooth interior surface and thin walls to disturb sediments as little as possible
- Hermetically sealed during ascent
- Allows subsampling
- Weight adjustable to penetrate various substrates
- Has a sufficient collection volume to meet analysis requirements
- Takes sediment samples efficiently and consistently in different depths of water
- Takes sediment samples efficiently and consistently at the sampling depths desired
- Does not contaminate or alter the nature of sediments
- Requires as little additional equipment as possible
- Is easy to use and reliable and does not require extensive training for personnel
- Is easily transported and set up on site

(Environment Canada 1994)

Generally, corers are used when samples are to be taken of the entire depth to be dredged. Sometimes sampling is done to a depth below the planned dredging depth; this is done to ensure that sediments with contaminant concentrations above those of overlying materials are not exposed and to characterize sediments that will be exposed if dredging depth is greater than expected.

Grab samplers are useful for surface sampling, when the properties of sediments are homogeneous (owing to a high accumulation rate, reworking by currents, ships, etc.) or if recurrent maintenance dredging is to be carried out. The different types of grab samplers are also easy to use and allow large volumes to be obtained more rapidly, which can be useful when carrying out ecotoxicological assays. On the other hand, samples obtained with grab samplers are usually disturbed to some extent (Baudo 1990, U.S. EPA/U.S. ACE 1998). When data quality objectives require a certain degree of sample integrity, the use of corers is preferable (ASTM 1997, Mudroch and Azcue 1995). In the presence of extensive natural or

man-made debris that may affect the performance of grab samplers, corers are also a better choice.

5.1.1 Corers

5.1.1.1 Applications

There are a number of different models of corers, each with different operating principles and adapted to different types of sediments or penetration depths (ASTM 1997, U.S. ACE 1996, Mudroch and Azcue 1995). A detailed description of the different types of corers in use can be found in a number of reference works (Mudroch and Azcue 1995, Mudroch and MacKnight 1991, ASTM 1997, Sly 1969, U.S. ACE 1996).

Most dredging projects on the St. Lawrence River involve dredging depths of no more than one metre. The techniques presented here generally allow samples to be taken to a depth of 3–5 metres, depending on the type of sediment. If greater penetration is required, drilling should be considered (U.S. ACE 1996).

5.1.1.2 Constraints

The main constraints associated with the use of corers in sediment sampling include (Blomqvist 1985, 1991; Parker 1991; Leonard 1990; Baudo 1990; Chant and Cornett 1991; Parker and Sills 1990; Mudroch and Azcue 1995):

- The loss of the surface layer of sediments at the moment of penetration due to poor draining of the water inside the corer or increased pressure on the cutting head, particularly during a high-speed freefall
- The resuspension of surface sediments inside the sample due to the use of a piston or by shocks transmitted to the sampler
- Reduction in core length due to internal friction or the use of a corer of insufficient diameter for the sediment type or penetration depth desired
- Repeated penetration of the corer due to the drifting of the vessel or strong suction of sediments during recovery
- The contamination of the lower horizons due to internal friction that pulls peripheral sediments inside the corer
- Washout of material from the bottom of the core during recovery due to the lack of a core retention system, excessive ascent speed or malfunction of the top valve.

The dimensions of the cutting head, body of corer and core tube are among the key factors to be taken into account to ensure high-quality samples. The parameters to be considered include:

- Cutting angle
- Diameter of corer
- Surface ratio (volume displaced by corer in relation to volume sampled)
- Internal friction ratio
- External friction ratio
- Length of core tube.

These parameters are described in section A.1 of Appendix A and shown in Figure A.1.

a) Composition and surface of core tubes. Tubes should be constructed of a chemically inert material such as:

- Stainless steel
- Aluminum
- Glass (internal liner only)
- Polytetrafluoroethylene (PTFE, Teflon®)
- High-density polyethylene (HDPE)
- Cellulose acetate butyrate (CAB)
- Polycarbonate (Lexan).

Tubes made of polyvinyl chloride (PVC) must not be used because they release phthalates and trace metals (Burton 1992, U.S. EPA 1994, U.S. EPA/U.S. ACE 1998, U.S. ACE 1996). Butyrate (CAB) tubes are also commonly used but are not recommended for the long-term storage of sediments since the material is slightly porous and allows moisture in the sample to seep out. New plastic tubes must be conditioned by soaking them in water for a certain amount of time.

Ideally, surfaces in contact with sediments should be smooth and without roughness to reduce internal friction. Owing to the manufacturing process, some plastic tubes have a grooved internal surface that may make subsampling difficult since grains of sand can get stuck between the tube walls and the piston used to extrude the core. Some tubes with a large

diameter (10 cm and over) of high quality have a fairly smooth surface but because of their high cost have to be reused.

b) Recovery of sample. The main product of the various factors affecting the sampler is poor sample recovery. Therefore, it is crucial that the sampler allow the release of excessive hydrostatic pressure that may build up in the top of the tube during descent and penetration and that may push out part or all of the sample.

Suction in the lower part of the corer during recovery is another important factor that can be countered by using a piston to create an internal vacuum, thus facilitating penetration. Some models may also be equipped with a closing system in the lower part of the corer (Appendix A, Figure A.2).

The shear strength of sediments must also be overcome so that the sample can be freed from the sampling surface. Several different techniques can be used to achieve this:

- Have the sampler rotate once or twice to cut through the sediments as it withdraws (however, the sampler should not rotate during penetration, in order to minimize sediment remobilization)
- Allow sampler to remain in place for a few minutes to allow the sediments to expand and adhere to the walls
- Use a core catcher (Appendix A, Figure A.2).

Using a closing valve or core catcher may result in the reworking of sediments, owing to the failure to release internal hydrostatic pressure and the reworking of surface sediments when the closing system opens.

5.1.1.3 Types of corers

Depending on their operating mechanism, corers can generally be divided into three main types (some corers can be modified to deal with hydrodynamic conditions and sediment grain-size distribution):

- Hand/mechanical corers
  - deployed by divers
  - deployed by a rod (piston)
- Gravity corers
  - open-barrel
  - piston/cable operated
- Vibratory corers

Standard operating procedures (SOPs) must be prepared prior to beginning field operations to ensure samplers are used safely and to maximize sampler performance.[1]

a) Corers deployed by divers. This approach has a number of advantages, including better sample quality in cores less than 2 m long (Appendix A, Figure A.3), since penetration conditions can be controlled (Wright 1980, Blomqvist 1985, 1990; U.S. ACE 1996) and an *in situ* assessment of penetration depth and conditions can be made. Divers can also describe the nature of the sediments and environmental conditions during both the site investigation and sampling stages. Dives must be made strictly in accordance with scientific diving standards. Push corers deployed by divers can only be used at depths no greater than around 30 m, owing to noncompression limits and the time required to take the samples.

Tubes are generally of small diameter (roughly 5 cm) and short (< 1 m). Larger tubes, up to 12 cm in diameter and 1.8 m in length, have been used successfully in the St. Lawrence River. They are inserted manually, using a specially adapted hammer as required. The tube is then closed with a stopper and sealed with an O-ring that can be adjusted by the diver. When the corer is removed from the sediments, the diver then inserts a second watertight stopper at the base of the corer to limit washout. A clamp attached to the corer is used to bring it to the surface with a winch installed on the boat.

Another type of diver-operated corer, the OCEI (Ocean Construction Equipment Inventory, U.S. ACE 1996) is sometimes used. It collects cores 3.8 cm in diameter and 76 cm in length and includes an internal piston and hammer to maximize penetration.

b) Corers deployed with a rod (piston corers). These corers are used from a fixed work platform (barge or boat). They are driven into the sediments using a rigid rod that allows the piston to be controlled by a line or second rod inside the main rod. Some models (e.g., Osterberg) also use pressurized water to drive the corer into the sediments. The use of rods limits sampling depth to less than 10 m, however (Wright 1980, 1991). External casing may be required to allow the corer to be used repeatedly in the same location by using extensions and cumulative sampling (Appendix A, Figure A.4).

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[1] Examples of SOPs are given in ASTM (1997), Mawhinney and Bisutti (1981), U.S. EPA/U.S. ACE (1998), Wisconsin Department of Natural Resources (1998), Mudroch and MacKnight (1991), Mudroch and Azcue (1995), Wright (1980, 1991) and Blomqvist (1990, 1985).

During sampling, the corer is lowered to the surface of the sediments and then driven into the sediment column by pushing on the rod or using a hammer. When the corer is brought to the surface and before the tube is taken out of the water, a plug must be inserted into the base to maintain the hydrostatic pressure that limits the loss of material. Different examples of this type of corer include the Butter, Osterberg (U.S. Dept. of Interior 1974), Swedish foil (Pickering 1965), Lowe-Acker stationary piston (U.S. EPA/U.S. ACE 1998), McClelland (Winterkorn and Fang 1975) and Livingston (Livingston 1955) corers and variants thereof. They are recommended for cohesive sediments (mud, clay) but not sandy sediments. In addition, operating this type of corer requires a certain amount of experience and an accurate knowledge of water depths in order to keep the piston just above the sediments.

c) Open-barrel corers. A number of models of open-barrel corers are also available (Appendix A, Figure A.5), all operating on the same principle. The bevelled cutting head at the base of the corer may be accompanied by a sediment retention system (e.g., eggshell core catcher), the use of which is optional. The upper part of the core tube is equipped with a valve-based closing system, which allows water under hydrostatic pressure to drain off during penetration and then hermetically seals the tube as the corer is hauled up. The valve can either be operated automatically or be spring activated. This type of corer, which is usually heavily weighted, requires a hoisting system to operate (winch and stand).

Although open-barrel corers are easy to use and readily available, they have a number of disadvantages, including shallow penetration depth, sampling artefacts depending on the sediment type, sampler repenetration[2] and a lack of control over penetration conditions. It is also recommended that ballast be increased rather than increasing the height of freefall, since the greater the freefall, the greater the chance of the corer not entering at a perpendicular angle to the bottom.

In addition, this type of corer does not work well in sandy sediments, which tend to dissipate the kinetic energy of the sampler, resulting in defective penetration.

Open-barrel corers are therefore recommended mainly for preliminary site investigations.

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[2] Repenetration occurs mainly in deep water during ascent and sometimes in shallower water when the boat is rolling and pitching.

d) Piston-operated gravity corers. Piston-operated gravity corers work on the same principle as rod-operated corers, except for the penetration method. The sampler must be lowered slowly until it touches the bottom, causing the tripping mechanism to release the corer, which penetrates the sediments after a short freefall of no more than 3 m (ASTM 1997). The length of the messenger line must be determined in advance so that the piston stops just above the water-sediment interface—usually 30-60 cm (U.S. ACE 1996). The length of the messenger line is determined with the following formula (U.S. ACE 1996):

$$L_t = V_t + L_c + F_c - (l_t - d_t)$$

where L_t = length of messenger line
V_t = vertical path of messenger
L_c = total length of corer
F_c = distance of corer freefall
l_t = total length of messenger weight
d_t = depth of messenger weight penetration in sediments

The various models allow cores of 6–10 cm in diameter and 3–15 m in length to be obtained, depending on the sediment type.

Since corers can weigh as much as 500 kg, an adequate hoisting device is required, providing sufficient clearance given the total length of the corer and messenger line. Once the corer is out of the water, the closing system at the base must be removed and the tube sealed. The liner can then be removed from the sampler. If required, the tube can be cut into equal sections or lengths corresponding to the subsampling interval. Sections are then sealed at the ends. Tubes can be used to store subsamples once the latter have been described and clearly identified.

One problem found in using this type of corer is incomplete penetration, when the piston does not extend completely into the corer. When the corer is being hauled out, traction on the hoisting cable pulls the piston to its full position, resulting in the suction of the sediments through the bottom of the corer until the corer clears the sediments. Using a split piston can solve this problem.

Another problem associated with this type of sampler is the abrupt movement of the piston during penetration that may result from the movement of the boat or the sudden

release on the tension of the cable during freefall. There is currently no recognized way of dealing with this problem (U.S. ACE 1996).

e) Vibratory corers. Vibratory corers (or vibro-corers) (Appendix A, Figure A.6) use high-frequency vibrations to displace a small ring of sediments into the pore space, thus reducing friction on the tube. The weight of the corer allows penetration to depths of 2–6 m depending on the sediment type and the centrifugal force of the motor. There are two main types of vibro-corers:

- In submersible (independent) systems (Rossfelder, Prych-Hubbell[3]), the motor is installed on the upper part of the core tube and is powered by electricity, compressed air or hydraulic pressure. The corer frame is lowered under water using a cable (Rossfelder 1999). Depending on the model, a second cable may be used to lower and raise the corer within the frame (Appendix A, Figure A.6). Generally, tubes are available in roughly 6 m lengths, which is ample for most dredging projects on the St. Lawrence River.
- In surface-operated systems (e.g. MetaDrill), the vibrator motor is supported by a vertical structure on the barge or boat (Appendix A, Figure A.6). The motor is lowered by vibration and/or a winch, which is also used to raise the corer after sampling. Depending on the depth, a string of rods allows the core tube to be lowered to reach the sediments. The advantage of the system is that the length of the core can be increased by adding extra tube sections.

The diameter of the cores is usually 6–10 cm. Some systems allow adapters to be used to accommodate larger tubes (12–15 cm in diameter).

Since vibro-corers are expensive, a number of systems have been developed using compressed air or commercially available electro-mechanical motors. The EPA has developed a corer based on a Wackerreg M3000 electro-mechanical vibrator attached to an aluminum tube, which weighs less than 20 kg in all and allows cores up to 2 m long to be obtained. Plans for a vibro-corer using compressed air are also available (Fuller and Meisburger 1982, Smith and Clausner 1993).

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[3] This corer was developed to collect sandy sediments up to 1.8 m deep in areas with currents as strong as 1.5 m/s. During ascent, the tube is automatically sealed with a sliding plate when the cutting head moves inside the contoured weight serving as the base of the corer (Prych and Hubbell 1966).

5.1.2 Grab samplers

5.1.2.1 Applications

Grab samplers are mainly used for studies not requiring the vertical characterization of the sediment column. Grab samplers are preferred in this context since they are easy to use and allow much larger volumes of sediment to be collected compared with corers.

Depending on the model used, grab samplers can collect sediments up to 30 cm deep. The penetration depth achieved by an individual device also depends on the nature of the substrate being sampled.

If the sediments are heterogeneous, it is strongly recommended that more than one type of grab be used (e.g. Ekman for soft sediments and Van Veen for coarse sediments) to improve sampling quality (Baudo 1990). This approach requires a good knowledge of the existing sediments, either from historical information or a preliminary site investigation.

The choice of an appropriate grab sampler depends on the type of materials to be sampled, degree of consolidation, speed of the current and the volume to be collected (Figure 5.1), taking into account specific data quality objectives.

5.1.2.2 Constraints

The main constraints of using grab samplers for sediment sampling are:

- Variability and poor reproducibility of depth of samplers depending on the sediment type, weight of bucket, speed at impact with sediments and incline (Environment Canada 1994, Blomqvist 1990, Baudo 1990, Downing 1984, Flannagan 1970). Ideally, performance should be documented using a camera (Blomqvist 1990) and a frame should be used to standardize sampling conditions (penetration and cutting angle).
- Variable sediment integrity depending on grab sampler used and type of substrate (Baudo 1990, Smith and Rood 1994, Mudroch and Azcue 1995). Transfer of samples from the grab to the homogenization container also results in a loss in sediment structure (ASTM 1997).
    - Reworking or movement of the surface layer caused by turbulence preceding grab as it touches bottom (Mudroch and Azcue 1995, Burton 1992, Blomqvist 1991).
    - Reworking of sediments caused by jaws pushing adjacent sediments into grab as they close.
    - Reworking of sample when grab straightens during recovery (if tilted during penetration).

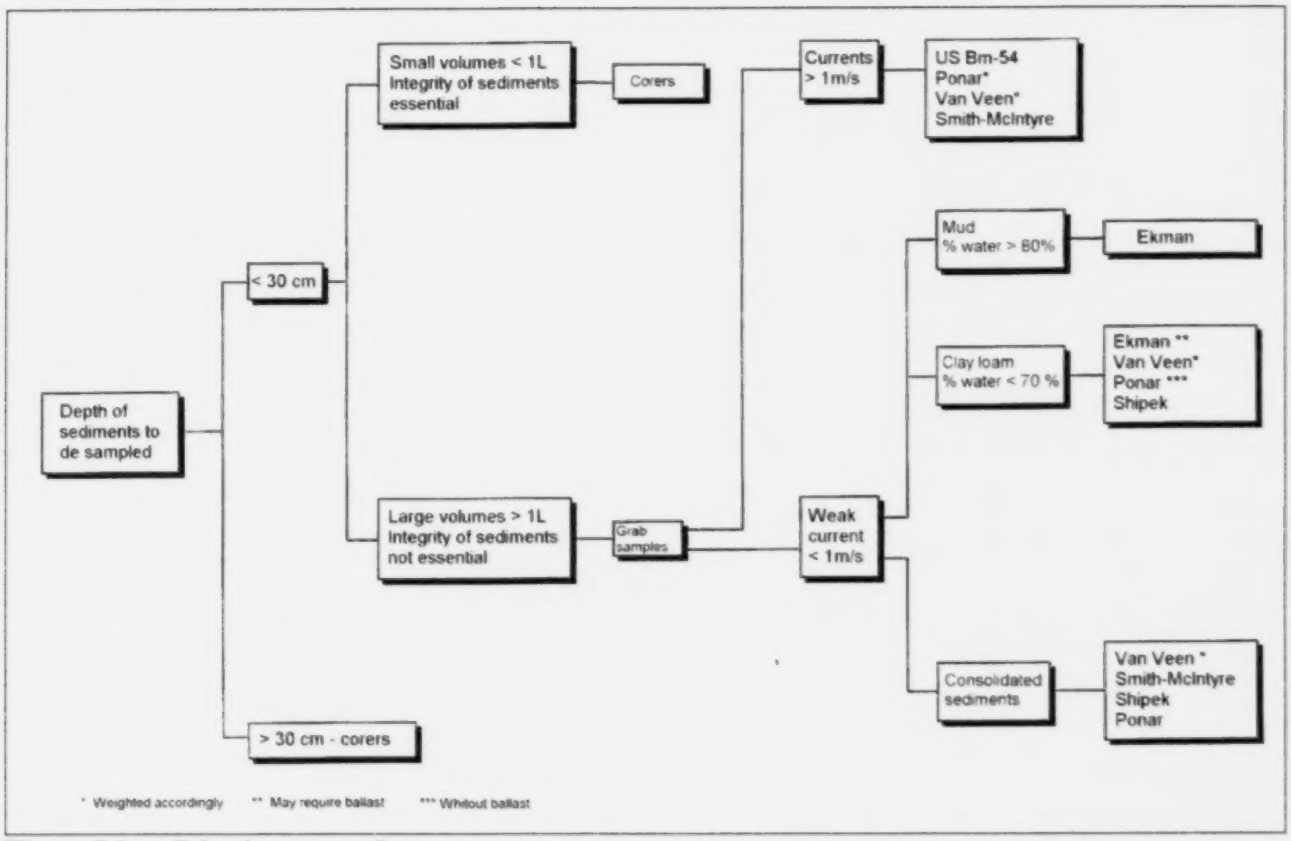


Figure 5.1 Selecting a sampler

- Incomplete closure of jaws resulting in a loss of sediments, which can be amplified by the ascent speed (Blomqvist 1990). Sediment reworking and washout can cause the loss of soluble compounds and volatile organic substances (ASTM 1997, Burton 1992).
- Contamination of sediments by metal or lining of sampler (paint, galvanization, corrosion; ASTM 1997), particularly in the case of trace metals. Most grabs, although they have a fairly old design, are now available in stainless steel; if not, a Teflon®, Kynar® (similar to Teflon®) or high-density polyethylene (HDPE) liner can be used as a satisfactory alternative in order to meet data quality objectives. If the sampler does not meet these requirements and the study design requires that metals be analysed, an aliquot can be taken directly from the centre of the sampler, avoiding the side walls.
- Difficulty in using grabs in a strong current. Under these conditions, some models have a tendency to tip over on the bottom. Using a heavier bucket or adding additional ballast may be required. In this case, a special hoisting system is required.

Nonetheless, grab samplers have a number of advantages:

- Allow a large volume of sediment to be collected, which is particularly useful in toxicological analyses.
- Several models allow subsamples to be taken directly from the grab itself (avoiding contact with the walls), which preserves sediment integrity fairly well. When the appropriate model is chosen, a grab can be used to sample almost any type of sediment: silt, sand, gravel or clay. The only exception is consolidated sediment.
- The possibility, with certain models, of adding weights to improve substrate penetration, to achieve the desired depth in accordance with the nature of the substrate.
- Protect samples fairly well from washout when the sampler is completely closed and the ascent speed is controlled.
- Commonly used and easy to obtain.
- Simple and quick to use, allowing for a number of samples to be taken in a short time.

5.1.2.3 Types of grab samplers

Since characterization studies for dredging projects often require large volumes of sediments (1–2 L, and up to 8 L for toxicity assays) for analysis purposes, only the larger models of grab samplers are suitable. Therefore, only large samplers that are particularly well adapted to sampling sediments in the St. Lawrence River and Gulf are described (Appendix B, Figure B.1). Two small-volume samplers are also described: the Shipek because of its versatility and high performance in different substrates and the US BM-54 for its ability to remain properly oriented in strong currents. The specifications for these various models can be found in Table 5.2. More detailed descriptions of the samplers can also be found in various sources (Environment Canada 1985, 1994; ASTM 1997; Mudroch and Azcue 1995; Burton 1992; Mudroch and MacKnight 1991; Rochon and Chevalier 1987; Håkanson 1986). Standard operating procedures for these samplers are described in detail in ASTM (1997).

a) Ekman. The Ekman sampler (Appendix B, Figure B.1) consists of a box-shaped bucket with a pair of jaws activated by two springs, in turn triggered by a messenger weight that is sent down the line from the surface. This sampler is particularly suited to sampling soft, silty or muddy sediments, and low-energy environments.

Table 5.2
Specifications for different types of grab samplers

Type	Dimensions (cm)	Depth of cut (mm)	Weight (empty) (kg)	Weight (full) (kg)	Volume (L)	Surface sampled (cm^2)
Standard Ekman	15 x 15 x 15	152	3	16	3.5	232
Tall Ekman	15 x 15 x 23	229	5	21	5.3	232
Large Ekman	23 x 23 x 23	305	7	38	11.9	522
Ponar	23 x 23	89	20	45	8.2	522
Van Veen	36 x 28	114	19	84	24	993
Shipek	20 x 20	102	61	69	3	400
Smith-McIntyre	31 x 31	40 in hard sediments	90	97–144	10-20	1000
US BM-54	56	50	46	Roughly 48	Roughly 0.2	

(Adapted from Downing 1984, Mudroch and MacKnight 1991, Mudroch and Azcue 1995, and Rickly Hydrological Co. 1999a)

- Advantages:
  - Available in several sizes
  - A frame can be added to increase stability
  - The machined scoops of the jaws overlap to minimize washout
  - Penetration can be increased by adding ballast
  - Flaps at the top open to allow subsamples to be taken directly from the bucket
  - The bucket's penetration angle (68°) ensures that the closing of the jaws does not result in the vertical movement of the sampler (Riddle 1989, Sly 1969).

- Disadvantages:
  - Requires an adequate hoisting system (winch and stand)
  - Given its wide footprint, the descent speed should be slow to reduce the shock wave that can disturb fine sediments and to avoid overpenetrating soft sediments
  - The strength of the springs is sometimes insufficient to close the jaws completely and the closing mechanism can become blocked by debris
  - Without a frame, the bucket can penetrate at an angle
  - When overpenetration occurs, adjacent sediments can enter by the top opening (Blomqvist 1990).

b) Ponar. The Ponar grab sampler (Appendix B, Figure B.1) uses two weighted jaws held open by a catch bar across the top of the sampler. The closing mechanism is activated when the sampler touches the bottom. During retrieval, the tension on the line keeps the jaws closed. Used widely because it is suited to many different types of substrates, the Ponar sampler is particularly adapted to sampling compact sandy and clay-loam sediments in low-current situations.

- Advantages:
  - The upper part of the sampler opens to allow direct subsampling;
  - Additional weights can be added to improve penetration.
- Disadvantages:
  - It tends to tip over on the bottom when the current is too strong
  - The descent speed must be controlled to reduce the shock wave, which can disturb fine sediments
  - The closing mechanism can become obstructed with debris
  - When the descent speed is too great, overpenetration may occur
  - Requires a hoisting system (winch and stand).

c) Van Veen. The Van Veen sampler collects sediments with two heavily weighted jaws, which have long arms extending beyond them to provide stability on the bottom (Appendix B, Figure B.1). There are several different models with different arm lengths and closing mechanisms. The model with long arms is usually preferable, since it provides a better cut (Riddle 1989). This model is particularly suited to sampling compact silty clay and sandy sediments.

- Advantages:
  - It can be used in strong currents (< 1 to 1.5 m/s)
  - A frame helps stabilize the sampler (Blomqvist 1991)
  - Depending on the model, the upper portion of the bucket can be opened, allowing subsampling to be done directly
  - Retains fine sediment well
  - Weights can be added to improve penetration
  - Of the grab samplers, it is the best suited to multiple situations and takes the most representative samples (ASTM 1997, Burton 1992).
- Disadvantages:
  - It has a tendency to tip over on the bottom despite the long arms

- It does not work well in consolidated sediments
- The descent speed must be controlled to avoid remixing surface sediments, despite the bucket design, which limits flow-through
- The closing mechanism can become obstructed with debris
- Overpenetration can occur in soft sediments because of its weight or if the descent speed is too great
- The jaws may close prematurely in rough water or if the descent speed is too great
- Requires a hoisting system (winch and stand).

d) Shipek. The Shipek grab sampler uses a semi-cylindrical bucket that cuts through the sediment and at the conclusion of a 180° rotation is held in a closed position by the action of two powerful springs (Appendix B, Figure B.1). Given the force exerted by the springs, precautions must be taken to ensure operator safety.

Like the Ponar grab, the Shipek can be used in most substrates. In addition, because of its mechanism, it is one of the only grab samplers that can be used to sample Champlain Sea clays. Advantages:

- Can be used in strong-current environments because of its heaviness and low centre of gravity
- The rapid rotation of the bucket cuts through the sediments well and reduces the reworking of the sample (Sly 1969)
- The bucket can be removed from the holding system, which allows subsamples to be taken directly from inside
- Once the bucket has been closed, it is completely protected from washout during ascent
- Two grab samplers can work in tandem (Sly 1969) to counteract the effect of rotation that may result in the tilting of the bucket in soft sediments.

- Disadvantages:
  - The speed of descent must be controlled to reduce the shock wave, which may disturb fine sediments
  - It collects smaller volumes than other grabs
  - Since the sampler is made of metal, samples may become contaminated (however, the bucket is available in stainless steel)
  - Requires a hoisting system (winch and stand)
  - Rotation may result in the bucket tilting in soft sediments.

e) Smith-McIntyre. The Smith-McIntyre grab consists of two jaws activated by very powerful springs (Appendix B, Figure B.1). It closes on the side rather than the bottom and

has a frame to increase stability. Although rarely used, this model has a very good design and is highly reliable (Downing 1984). It is particularly suited to gravel, sandy and clay-loam sediments. Given the force exerted by the springs, precautions must be taken to ensure operator safety.

- Advantages:
  - Can be used in either low-energy or strong current environments
  - Is effective in hard sediments because of the powerful spring closing mechanism
  - Has a holding system with large feet which adds stability on the bottom and keeps the bucket from digging in
  - Weights can be added to increase penetration and stability in areas of strong currents
  - Soft rubber flaps reduce washout during ascent
  - Can be used in difficult conditions because of its heft and stability.

- Disadvantages:
  - Overpenetration may occur in soft sediments because of its weight or if the descent is too fast
  - Because of its design, must be lowered slowly to prevent the remobilization of surface sediments
  - Metal grab can contaminate sample (is also available in stainless steel)
  - Jaws can become stuck and result in washout of fine sediments
  - Requires a hoisting system (winch and stand).

f) US BM-54. The US BM-54 grab sampler has an elongated shape and a stabilizing fin (Appendix B, Figure B.1). It works on the same principle as the Shipek grab. It is used for sampling fine to moderately compact river sediments (Rickly Hydrological 1999), and is also very effective in strong currents. The body of the sampler is made from cast steel although the bucket may be made of stainless steel.

- Advantages:
  - Stabilizing fin ensures consistent orientation in current, ensuring stability during descent
  - Prevents washout during ascent.
- Disadvantages:
  - Descent speed must be controlled to reduce shock wave, which may disturb fine sediments

- Allows only a small volume of sediments to be sampled at one time, so repeated sampling is usually required
- A winch is required because of the weight.

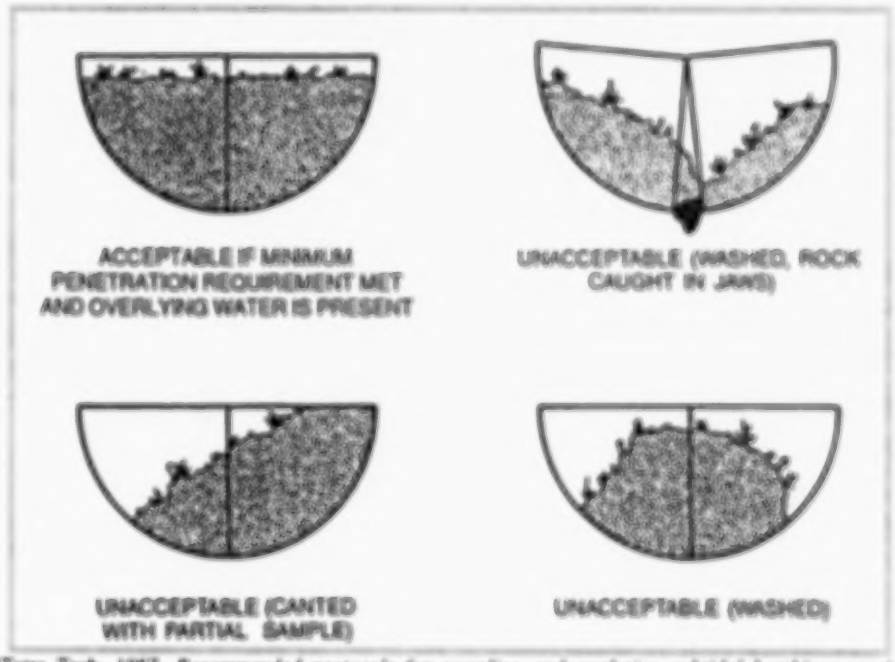
5.1.3 Acceptability criteria of samples

If a sample is rejected because it does not meet data quality objectives, a new sample must be collected, preferably upstream of the first sample. Similarly, a partial sample that does not meet data quality objectives must be replaced by a satisfactory sample taken subsequently. If conditions limit sampler performance, the sample can be a composite of several grabs that, individually, do not meet all quality criteria. The problems encountered should be described in the field notebook.

The criteria for the acceptability of a grab sample are:

- The overlying water is clear or not excessively cloudy
- Penetration depth was reached (sufficient volume)
- The bucket was closed completely.

Figure 5.2 shows several reasons for rejecting a grab sample.


ACCEPTABLE IF MINIMUM PENETRATION REQUIREMENT MET AND OVERLYING WATER IS PRESENT
UNACCEPTABLE (WASHED, ROCK CAUGHT IN JAWS)
UNACCEPTABLE (CANTED WITH PARTIAL SAMPLE)
UNACCEPTABLE (WASHED)

(Tetra Tech, 1987, *Recommended protocols for sampling and analyzing subtidal benthic macroinvertebrate assemblages in Puget Sound*, p. 20, with permission)

Figure 5.2 Conditions of acceptability of a sediment sample taken by grab sampler in a characterization study

The criteria for the acceptance of a core sample are:

- Sampler penetration is sufficient and adequate (vertical) based on the dredging depth and data quality objectives (a greater penetration depth may be required to characterize sediments exposed after dredging)
- The recovery rate (ratio of length of corer-to-penetration depth) is sufficient and the sample volume is acceptable
- Sample integrity is maintained (the sample quality is more important for ecotoxicological analyses than for physicochemical characterizations)
- The sample is representative (sampling of entire sediment column without loss of sediments).

5.2 EXTRACTION OF PORE WATER

Pore water, which fills the interstices between sediment particles, acts to create a bond between sediment particles and the overlying water. In general, concentrations of different substances tend to reach an equilibrium between the aqueous and particle phases. This equilibrium can be modified by various physicochemical factors. Pore water can therefore be used to assess the effect of sediment contamination on aquatic organisms, since it reflects toxicity better than whole sediments or eluviates (Burtflap and Allen 1995, Adams 1991, Ankley et al. 1991, Giesy and Hoke 1990).

5.2.1 Constraints

All sampling methods modify, to varying degrees, the chemistry of pore water (ASTM 1997). The resulting modifications vary enormously from study to study depending on the nature of the sediments, and the specifics of the sediment sampling, extraction and preservation methods used. The compounds most likely to be modified by extraction method are dissolved organic carbon, dimethyl sulfide, ammonium hydroxide and the main cations. Furthermore, volatile compounds are usually affected by sampling and storage since volatile and semivolatile substances decrease substantially after storage for five to seven days respectively (ASTM 1997).

Such modifications are caused mainly by (ASTM 1997, Mudroch and Azcue 1995, Carignan 1994, Bishop et al. 1970):

- Oxidation (changes in oxidation-reduction conditions, AVS losses)
- Temperature increases

- High pressure (addition of metabolites)
- Contamination by materials used for sampling and extraction, particularly filters.

Sediment oxidation is probably the most important disruptive effect. Given the generally anoxic nature of sediments, all steps in the processing of the sample, both in the field and laboratory, must be done in an inert (nitrogen or argon), controlled-temperature atmosphere (Burton 1992, ASTM 1997). The use of a grab sampler may favour sediment oxidation. Therefore, using a corer for bioassay samples can be more appropriate.

Pressure and temperature variations inevitably affect the distribution of certain chemical components of pore water (Mudroch and Azcue 1995). Some extraction methods entail the application of high pressure to sediments to extract the water. This may damage the cells of microorganisms in the sediments or result in chemical changes to the pore water.

Furthermore, the use of a filter is not recommended for studies of trace metals since such metals become concentrated on solids such as filters (ASTM 1997). Filtration reduces the toxicity of pore water (Ankley et al. 1991).

Lastly, the separation of pore water from sediments in the laboratory does not always provide results that are representative of the nature and chemical properties of the pore water (Environment Canada 1994). Nevertheless, by taking the necessary precautions (including controlled temperature; prevention of oxidation; appropriate choice of sampling, extraction and preservation methods and materials; and reduction of storage time), sampling artefacts can be minimized in order to obtain pore water that is the most representative possible.

5.2.2 Pore water extraction methods

The selection of an extraction method depends mainly on sample volume and time constraints, which are determined by the study objectives. The method used must also minimize changes to the sample caused by oxidation, temperature variations, filtration and contamination by the sampler.

There are four main methods for extracting pore water (Figure 5.3).

- *In situ* methods:
  - dialysis
  - direct suction.

- Indirect methods (in the laboratory):
  - centrifugation
  - squeezing.

In situ methods are generally recommended because they help prevent changes to the sample caused by temperature, pressure and oxygen. Their main disadvantage is the small volume of pore water produced, mainly because of the long length of time required for equilibration in dialysis and suction in the direct suction method. In addition, equilibrium may not be obtained because of the limits of diffusion in sediments and the ability of membranes to absorb dissolved elements (Grigg et al. 1999, Environment Canada 1994). *In situ* methods, therefore, are best suited to small volumes (less than 0.5–1 L).

Indirect methods, which involve the extraction of pore water from sediments previously collected, cause more disturbance to the sample. The main artefacts of these methods include the exposure of pore water to oxidation and increased temperatures and chemical selection by filters. Such methods are preferable, however, for obtaining the large volumes (1–3 L) required for toxicity assays.

If bioassays are planned, special containers designed to promote phase equilibrium can be inserted in sediments *in situ* or in a microcosm containing sediments taken in the field and transported to the laboratory.

Sections 5.2.2.1 and 5.2.2.2 provide a brief overview of these methods, particularly the main logistical and analytical constraints involved in extracting pore water. There are currently no standard operating procedures for the different methods, which are constantly evolving, with many authors proposing various modifications and adaptations to solve the problems encountered (ASTM 1997). The advantages and disadvantages of these pore water extraction methods are shown in Table 5.3.

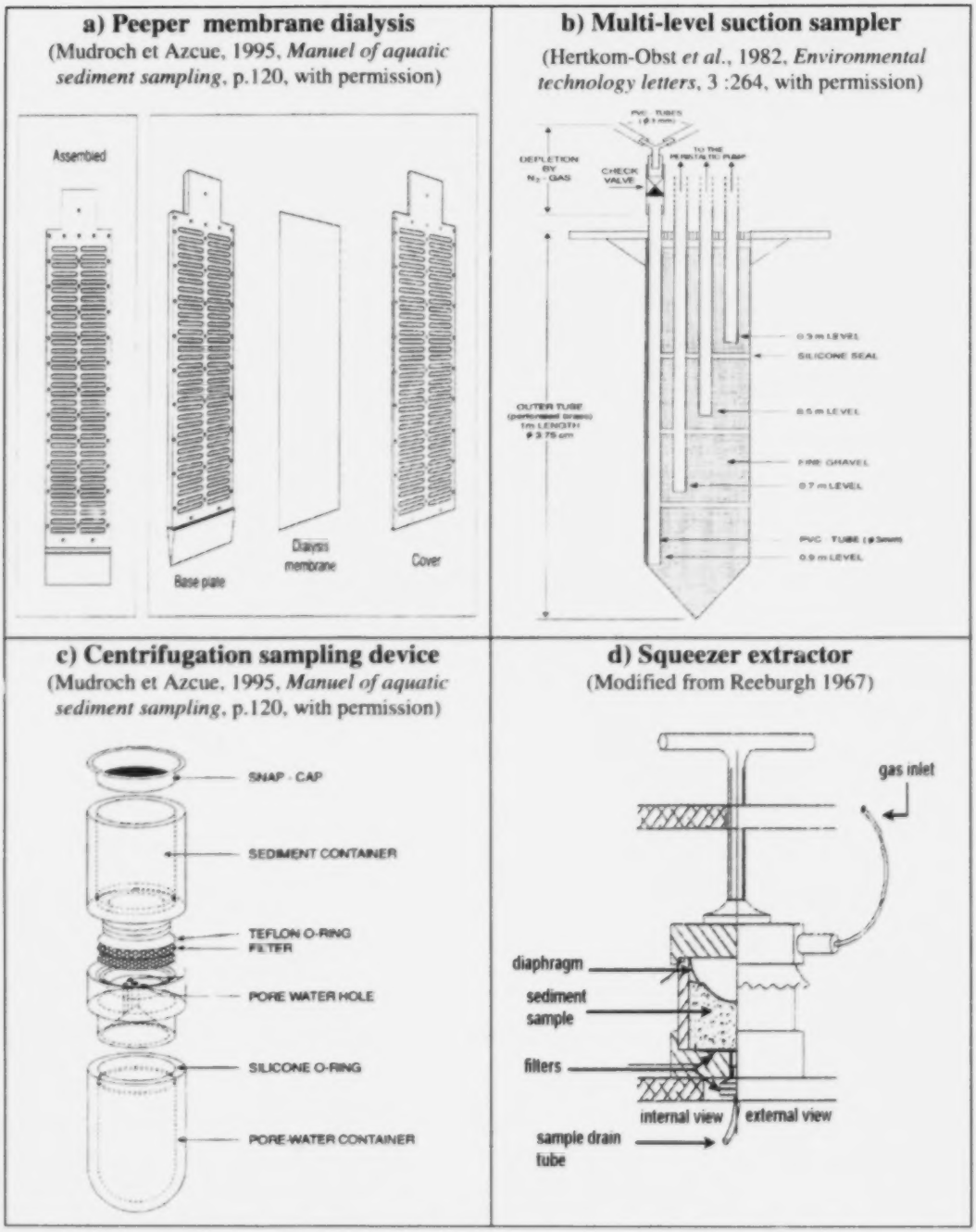


Figure 5.3 Schematic of pore water extraction systems

5.2.2.1 In situ methods

Dialysis. Dialysis is based on the principle of the diffusion of dissolved substances through a selective membrane that separates the deionized, deoxygenated water in the sampler from the pore water. The composition and size of the membrane pores varies, providing a certain selectivity in terms of solutes.

Recommendations on using dialysis for pore water extraction

- The device must be purged with an inert gas for 24 to 48 hours (for up to 30 days according to Carignan [1984]) and protected from the ambient air until it is installed.
- Divers usually insert the device in the sediments.
- The water in the sampler must be allowed to equilibrate with the sediment interstitial water. The required time for equilibration has been reported to be between 15 and 20 days, or must be determined experimentally (Carignan 1984).
- Subsampling is usually done with a syringe or tubes attached to the device, and must be done quickly to avoid modifications from temperature changes or oxidation-reduction conditions.
- A polysulfone membrane must be used (Butfflap and Allen 1995). Some polymers are inappropriate because they can absorb certain nonpolar compounds (ASTM 1997). Cellulose membranes are inadequate because they decompose too quickly (ASTM 1997). They can lead to the under- or over-estimation of the concentrations of the various components of pore water (Carignan 1984).

Volume obtained. In general, several dozen millilitres can be obtained after equilibration but the volume depends on the type of device used (Table 5.4).

- Bag- or cylinder- (*stacker)* type devices handle the greatest volume of water and should be favoured (Table 5.4).
- Peepers (Table 5.4), which collect only small volumes (3–5 mL per cell), are mainly used for geochemical profiles (Azcue et al. 1995).
- The VESPOS is a peeper-type sampler that has equilibration chambers that may hold up to 60 mL (Azcue et al. 1995). It requires a longer equilibration time, however.

Table 5.3
Advantages and disadvantages of pore water extraction methods

Method	Advantages	Disadvantages
Laboratory		
Squeezing	- Simple, portable equipment - Inexpensive - Sediment composition available - Provides large volumes	- Possible oxygen contamination - CO_2 degassing, which changes pore-water composition - Elevated temperatures - Pressure-related additions of metabolites
Centrifugation	- Simple - Sediment composition available - Provides large volumes	- Risk of sampling artefacts - Pressure artefacts - Possible changes due to oxidation and elevated temperatures
In situ		
Dialysis	- Simple, economical design and operation - No induction of interstitial water flow - Allows maximum replication - Analysis of dissolved gases is possible - Temperature-, pressure- and oxidation-related artefacts are avoided Certain membranes can be used to provide solute selectiveness	- Disturbance of the sediment structure - Need for scuba divers - Lengthy (minimum 12 days) - Equilibration chamber required - Risks of incomplete equilibration (equilibration time ideally should be determined experimentally since it is variable) - Dialyzer and circuit must be purged with nitrogen gas - Risks of microbial attack of membranes when cellulose membranes are used - Development of electrical potential through membrane
Direct suction	- Simple and easy to use - Closed system minimizes contamination risks - Stable - Allows sampling at well-defined depths	- Fine particles can be collected - Oxidation effects are hard to prevent completely - At a given depth, only small volumes can be collected

(Modified from Mudroch and Azcue, 1995)

Table 5.4
Main types of dialyzer samplers

Type	Volume obtained	Characteristics
Bag type		
Method developed by Mayer (1979)		Bag or container filled with distilled water; chemical equilibration with the pore water is carried out through a dialysis membrane. Sampling at several depths a possibility (several bags in Lucite® tube)
Höpner (1981)	3 mL per chamber	Sampling at several depths (20 sampling chambers)
Bottomley and Bayly (1984)	10–12 mL per chamber	Modification of Mayer's method (1979) Sampling at 10 different depths using several bags in a perforated, covered Lexan sleeve
Peeper type		
Hesslein (1976)	3–5 mL per chamber	Sampling chambers machined in a rectangular acrylic panel. The water enters through a membrane covering the entire panel, held in place by another panel of the same size. The latter has slots cut in the same position as the compartments.
VESPOS, Azcue et al. (1995)	Up to 60 mL per chamber	Automatic closing of containers with pore water before sampler is recovered

Direct suction. If this method is used, removal of the pore water from unconsolidated sediments is done after filtering, under hydrostatic pressure or by suction using long-needle syringes or a tube. Divers can also be used if the depth warrants it.

Suction systems vary according to the type of filter used, location of filter on the sampler, suction mechanism and whether or not sampling can be carried out simultaneously at several depths. Table 5.5 describes the different techniques that are used for the direct suction of pore water. For more details on these methods, see Mudroch and Azcue (1995).

The main disadvantages of this method are the small volumes obtained, use of filters that may introduce sampling artefacts and the complexity of models described in the literature.

- Direct suction is not recommended because of the logistical problems involved in using a pumping apparatus and the time required for equilibration and pumping to obtain sufficient volume. Direct extraction using a syringe is easy, but not recommended because it only produces very small volumes.

Table 5.5
Techniques used for direct suction of pore water

Sampler	Volume obtained/time	Characteristics
Single-depth sampling		
Zimmerman et al. (1978)	Variable depending on sediment type	The water is extracted using a ceramic filter at the end of a tube and then recovered with a vacuum pump.
Barnes (1973)		Stainless steel filters in 3 thicknesses; water collected in a stainless steel tube.
Goodman (1979)		Probe with 3-mm diameter and filter with 0.45 mm mesh; suction using a syringe connected to probe with a tube.
Howes et al. (1985)	5–10 mL at a time	Glass tubing with Teflon® sleeve; direct suction with syringe; precise depth interval measured uncertain.
Rey et al. (1992)		Modified from Zimmerman et al. (1978); uses a polyethylene filter.
Multiple-depth sampling		
Sayles et al. (1973)		Stainless steel tubing with five filter-covered sampling ports (Teflon® filters); suction using an instrument functioning as a large syringe with a spring-loaded cylinder providing the suction; overpenetration of the sampler into the sediments could result in incorrectly assigned depths; possible contamination with overlying water.
Murray and Grundmanis (1980)		Modification of technique developed by Sayles et al. (1973) for sampling pore water and gases.
Montgomery et al. (1981)	Equilibration for 3 days, 4 mL/min	For shallow depths (0.5 m); allows repeated sampling in an anoxic environment; simultaneous sampling from four sediment depths in a Plexiglas tube with four independent chambers with Teflon® filters; water suctioned off with vacuum pump at a flow rate of 4 mL/min.
Brinkman et al. (1982)	Equilibration for 1 day, 5–10 mL	Based on technique developed by Goodman (1979); probes equipped with paper or fibreglass filters on a frame; depths sampled chosen according to length of a variable tube making up part of the probe; metal plates on the legs of the frame prevent the sampler from sinking into the mud.
Herkorn-Obst et al. (1982)	400 mL/h	Sampling using PVC tubes equipped with filters, at four different depth levels in a perforated brass cylinder. Pore water is withdrawn with a peristaltic pump.
Watson and Frickers (1990)	Equilibration for 2 days	Sampling by applying a vacuum to chambers linked, through porous segments, to successive depths in sediment profiles.
Buddensiek et al. (1990)	Equilibration for 1–2 weeks	Repeated sampling at exact depths at 1 cm intervals up to 35 cm deep; stable unit that can be operated year round.
Hursthouse et al. (1993)	5 mL/2 min	Technique similar to that used by Watson and Frickers (1990), except with filters arranged in a spiral sequence in a polyethylene tube; suction with a hand pump.
Bertolin et al. (1995)		Allows samples to be taken without removing the device (allowing replicates); samples at greater depths than the models developed by Watson and Frickers (1990) and Hurshouse et al. (1993).

5.2.2.2 Indirect methods

Centrifugation. This method consists of centrifuging sediments to recover and filter (in some cases) the supernatant.

Recommendations for extracting pore water by centrifugation. (Source: ASTM 1997, Mudroch and Azcue 1995, Environment Canada 1994, Carignan et al. 1985).

- Use a glove box to handle and centrifuge sediments.[4]
- Put homogenized sediments in polycarbonate, Teflon® or Corex centrifuge tubes to prevent contamination. Stainless steel tubes can also be used when contaminants are strictly organic in composition.
- The recommended centrifugation time is 30 minutes for 10 000 g (ultracentrifugation) in order to extract more water from the samples and to remove colloidal matter and non-dispersible clays. Slower centrifugation generally requires subsequent filtering to remove these substances.
- Filtration may cause significant changes in the toxicity of pore water (Ankley et al. 1991) because of the selective adsorption of trace metals and some nonpolar compounds (Ankley and Thomas 1992, Burton 1992, ASTM 1997); in addition, unfiltered samples must be tested to determine if there has been any contamination from the filter. Chemical concentrations of substances found in pore water can vary according to the type (polycarbonate, Teflon®, etc.) and size of filter used (ASTM 1997, Mudroch and Azcue 1995). Filters therefore must be chosen wisely.
- The temperature of the sediments during centrifugation should reflect the *in situ* temperature to prevent chemical modifications.
- Siphon off or decant supernatant, filter if necessary and keep in pre-treated containers.
- Pretreat filters by soaking them (as appropriate) in deionized water for 24 hours (48 hours for nylon filters).
- Extract pore water within 24 hours after the sample was taken.
- Re-centrifuging the sample is recommended before it is tested or frozen, to eliminate suspended particles (Carr and Chapman 1995).

Any delay in implementing, or deviation from, these recommendations should be considered as a possible source of chemical modifications to the pore water sampled.

Volume obtained. Roughly 400 mL per litre of sediments.

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[4] Saager et al. (1990) developed a method that does not require the use of a glove box because the filter is integrated with the centrifuge tube. It allows 75% of the total water content of sandy sediments to be recovered, compared with only 25–30% by squeezing.

Squeezing. This method involves squeezing sediments through filters to extract the water. The type of filter used may vary, depending on the technique (Table 5.6).

Recommendations for extracting pore water by squeezing

- Sediment handling, squeezing and filtration must be done in a glove box with controlled temperature and atmosphere to prevent chemical modifications.
- To prevent contamination, surfaces that come in contact with sediment should be made of inert materials such as Teflon® or Corex. Stainless steel may also be used if the contaminants in question are organic.
- The temperature of the sediments during squeezing must reflect the *in situ* temperature to prevent chemical modifications.
- When a filter is used, a sample of unfiltered water (obtained by ultracentrifugation) should also be tested to verify possible contamination by the filter.
- Pretreat filters by soaking them (where appropriate) in deionized water for 24 hours (48 hours for nylon filters) (Carr and Chapman 1995).
- Pore water must be extracted within 24 hours of sample collection.
- The pressure applied on the sediments should be determined experimentally to avoid the introduction of metabolites from the breakage of the cells of micro-organisms or debris (Bolliger et al. 1992). Excessive pressure may result in the introduction, to various degrees, of Na, K, HPO_4, H_4SiO_4, NO_2 and dissolved organic carbon, which may increase their concentrations (Mudroch and Azcue 1995).

Volume obtained. The volume obtained is usually a few dozen mL per hour. The total volume is a function of the volume of filtered sediments and the squeezing technique used (Table 5.6).

Any delay in implementing, or deviation from, these recommendations should be considered as a possible source of chemical modifications to the pore water sampled.

Table 5.6
Characteristics of various techniques for extracting pore water by squeezing

Method	Volume obtained and squeezing time	Filter	Characteristics
Presley et al. (1967)	40% of total water content	0.4-mm Millipore	Teflon® lining prevents contamination; gas pressure of 70 kg/cm^2
Reeburgh (1967)	25 mL/30–45 min		Gas pressure on a plastic diaphragm; no metal pieces
Kalil and Goldhaber (1973)	10 min to 1 h	Filter paper	Thermoregulated pressure (3 tonnes); plastic lining
Sasseville et al. (1974)	400 mL*	Filter paper, followed by refiltering through a 0.45-mm Millipore filter	Piston squeezing in PVC cylinder
Robbins and Gustini (1996)	10–50 mL*		Adaptation of device developed by Reeburgh (1967) for unconsolidated fine sediments
Matisoff et al. (1980)		0.22-mm Millipore	Nylon squeezer, gas pressure of 3.4 atm applied against a rubber diaphragm
Bender et al. (1987)	30–60 min per 2 cm of core	10-mm polyester	Pressure on surface of core using Lucite® piston with a hole through the centre containing a filter; manual squeezing; chemical changes in water due to reactions between solid and liquid phases
De Lange (1992)		0.2-mm cellulose-acetate	Modification of device developed by Reeburgh (1967); made of Derlin®
Rosa and Davis (1993)	Time-consuming compared to others		Multiple clamping device; gas pressure
Jahnke (1988)			Extraction of pore water from a core at defined thicknesses
Petryk and Dustin (1993)	25% in 30 min	Paper	Gas pressure; modified shape of pressure-applying surface provides increased sampler efficiency

* Assuming 50% recovery.

5.3 GENERAL FIELD PROCEDURES

These are general points that must be taken into account when carrying out a field campaign.

- Sample collection and handling methods must be defined before the sampling campaign begins. Any deviation from standard operating procedures (SOPs) must be clearly identified and recorded in the field notebook.
- All the sampling and measurement equipment must be calibrated and checked before and during the field campaign. Tests and calibration must be documented and recorded before and during the campaign.
- All equipment must be clean and ready for use. The inside of the sampler must be cleaned in accordance with analysis requirements.
- Two anchor lines are required to keep the boat still. Stability is crucial to maximize sampling performance and ensure accurate positioning.
- The site should be approached from downstream to avoid disturbing the sediments at the site. Turbulence created by boat engines may cause the reworking of soft sediments to depths of up to 6 m (Sly 1969, Blomqvist 1990).
- A stainless steel homogenization container large enough to hold the entire sample must be on hand.
- All sample containers and covers must be marked with the sample number, date and type of analysis required (organic or inorganic) before sampling.
- The hoisting cable must be attached to the sampler with a swivel hook to limit kinking during descent. During ascent, the grab must not be taken completely out of the water until a container filled with water has been placed under the sampler to maintain the hydraulic head. Similarly, corers should not be taken completely out of the water without putting a stopper on them. A decrease in the hydraulic head can result in the rapid loss of the sample.
- Once the grab or corer is in the boat, care should be taken not to shake it in order to prevent the remixing of surface sediments. Rinse the outside of the grab and corer, paying attention to potential washout of the sediments inside. Cleaning the sampler in this way minimizes the risks of cross contamination.
- Representative samples must be taken at all stations to meet study objectives.
- After measurements are taken, the sample is logged and described and subsampling is carried out, in accordance with the data quality objectives set for the study.
- Field information (description of samples, sampling conditions, sampler performance) must be recorded in detail, completely and painstakingly. Even information thought to be unimportant when observed may turn out to be important later.

- The recording of all samples must be consistent and include information on identification, and the nature and destination of the samples. The loss or misidentification (error, loss of label) of a sample may affect the interpretation of the results.
- During sampling, replicates must be taken as often as deemed necessary.
- For replicates, the boat must be moved a small distance to avoid sampling sediments already disturbed by sampling operations.
- The sampler must be cleaned before moving on to the next station.
- During the sampling campaign, the field team must be on the lookout for potential sources of contamination, including such things as exhaust fumes from the boat motor, surface of transport vehicles (back of a truck), dirty samplers, and the working surface in the boat. During sampling and handling of samples, the team must not eat or smoke near the samples.

Standard operating procedures must be formulated to ensure data quality objectives are met. SOPs must describe all steps and operations to be carried out by the field team, taking care to describe the operation of the various samplers and measuring devices to ensure that measurements are precise, reproducible and accurate.

A checklist to be used in formulating standard operating procedures can be found in Table C.1 of Appendix C.

5.4 POSITIONING METHODS

The co-ordinates of sampling stations must be determined before sampling activities begin. Sampling must be done as close as possible to previously identified locations, inasmuch as the station generally represents a given surface area. During collection, the co-ordinates of the sampling location must be recorded.

Positioning requirements for dredging projects include:

- Minimal precision < 1 m
- Real-time positioning capacity
- Output of positions in the form of geographic co-ordinates (latitude, longitude, reference system, datum) to facilitate conversion to other co-ordinate systems
- Minimal effort required for data processing
- Minimum updating of co-ordinates every three seconds
- Ability to store co-ordinates in memory
- No interference with shipping or natural structures nearby
- Complete coverage during the entire day and minimum accuracy ensured.

5.4.1 DGPS positioning

Satellite-based global positioning systems (GPS) are probably the most appropriate for determining station position quickly and accurately. Until recently, the initial accuracy of GPS was around 50 to 100 m. Different data acquisition techniques were thus used to achieve accuracies of around one metre or less.

For example, measurements can be improved by obtaining a differential correction signal from a second receiver positioned at a known point or from the Canadian Coast Guard's network of beacons (http://www.ccg-gcc.gc.ca/dgps/main_a.htm). The Coast Guard's beacon network[5] allows users to obtain accurate positions without having to use a second unit and extra communications system.

The network of beacons emitting the correction signals covers the entire St. Lawrence River and Gulf of St. Lawrence (http://www.ccg-gcc.gc.ca/dgps/main_a.htm). After May 2000, satellite signals were no longer scrambled, resulting in the greatly improved positioning accuracy of standard GPS systems. Consequently, standard systems (without DGPS) are now sufficient for sampling work in dredging projects in most cases.

Despite the good performance of satellite positioning systems, it is crucial in the field to:

- Verify the reception quality of the signal during positioning
- Record information on signal quality in the field notebook (HDOP value)
- Determine whether positioning needs to be verified with an alternate method
- Use static positioning (which takes an average of positions over several minutes) rather than instantaneous positioning.

The location of stations must be detailed enough that they can be found again if necessary. In addition, geographic information (location, toponymic identification of sites), DGPS co-ordinates (with datum and signal quality index, static or instantaneous mode) and, where required, the positions of conspicuous beacons (poor-quality GPS positions are often observed in data processing) must be described.

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[5] Differential corrections provided by the Canadian Coast Guard are in acordance with the North American Datum of 1983 (NAD83). DGPS positions obtained must refer to the same datum.

5.4.2 Alternative methods

Because of constraints related to satellite availability and signal quality, an alternate system may have to be used. For example, tall structures on docks may limit satellite reception. In this case, employing surveying techniques using a geodetic marker, generally present on this type of marine structure, is suggested. Using chaining and a dummy compass or a total station allows adequate positions to be obtained (accuracy of around one metre).

Other alternative methods are described in various training manuals and documents (Environment Canada 1994, Mudroch and MacKnight 1991, Mudroch and Azcue 1995, U.S. EPA 1994).

6 Handling Samples

6.1 SEDIMENTS

6.1.1 General recommendations

- All samples must be handled in accordance with the quality assurance and control program and data quality objectives. At each step, the sample must be handled using a systematic approach and work should be done in a space that is clean, sufficiently large and protected from external contamination sources.
- Safety rules in the field (jobsite safety, handling of contaminated sediments, and management of toxic products such as solvents and acids) must be followed (see chapter entitled "Health and Safety in the Field").

6.1.2 Recovery of sediment cores

- During recovery, the base of the corer must be sealed with a stopper before the tube is completely withdrawn from the water.
- Penetration depth must be measured before cleaning the corer.
- The core tube should be cleaned (when applicable) to measure the length of the core. If the core must be preserved for a period of time before subsampling, the general description of the core must be completed immediately. Photographing the core is recommended.
- Core tubes must be sealed with leakproof stoppers (O-ring, electrical tape) to prevent sediment loss. The upper part of the corer should be kept full of water to prevent the remixing of the surface sediments. Because of turbulence, an insufficient amount of water often causes more disturbance than the complete absence of water.
- Before subsampling, cores must be allowed to sit for a time to allow any remobilized sediments to settle. The overlying water can be siphoned off, taking care to avoid disturbance of the sediment-water interface. If disposable liners are used, holes can be punched at regular intervals to drain the water from the top part of the core.
- Sediment cores must be kept in an upright position to await subsampling.

a) Subsampling. The subsampling of sediment cores can be done in the field or in the laboratory. In either case, the compatible subsampling instruments must be used (stainless steel, Teflon® coating, polyethylene, etc.) and adequately conditioned for the type of analyses planned.

The subsampling interval must be established beforehand in accordance with the quality objectives of the project (for dredging projects, the interval is usually 30 cm or more). At a minimum, two subsamples must be taken to characterize the surface and deep layers.

Depending on the type of core tube used and subsampling approach chosen, cores can be extruded vertically or horizontally, sectioned laterally or cut in sections of defined length. If tubes are to be reused, extrusion is the only method that can be used.

Extrusion

- Must be done quickly because of the consolidation of the sample on the walls of the tube (ASTM 1997).
- Usually done with a piston which is held vertically with a frame. The piston is inserted in the bottom of the corer. The corer is then lowered onto the base using a threaded rod, with a given number of turns corresponding to a given thickness (e.g. 4 turns = 1 cm). An alternate method is to push a piston (manual or hydraulic) into the corer, which remains fixed. For longer cores (> 1.5 m), horizontal extrusion using a piston is often the most practical. Hydraulic extrusion systems using water pressure are generally used for corers with a diameter smaller than 10 cm. When larger diameter corers are used, the weight of the sediment inside exerts a strong pressure, so that the water infiltrates around the piston through the grooves made by the extrusion of the tubes.
- If sediments are sufficiently compact, horizontal extrusion can be done on a flat surface or preferably an elongated tray. Since subsampling for dredging projects requires the homogenization of an interval of at least 30 cm long, the poorer precision that is obtained with horizontal extrusion will have minimal effects on the quality of the results.
- The subsampling interval must be great enough to minimize the washout of sediments during sampling and extrusion. If subsampling intervals of around one centimetre are envisaged, the outer layer of sediments along the edge of the tube should not be sampled since this will bias the results, particularly if pronounced gradients are present (Chant and Cornett 1991). The use of a specially designed adapter tailored to the diameter of the corer is recommended. This allows a roughly 2–3 mm outer layer of sediment of material in contact with the tube to be discarded in a saucer (Mudroch and MacKnight 1991, Mudroch and Azcue 1995).

Transverse cutting. If disposable tubes are used, cores can be subsampled directly from the tubes after they have been cut. This is only feasible if the sediments are compact enough (< 50–60% water). Sometimes, the upper part of the core has to be extruded since the sediments are too watery.

- The tubes are cut longitudinally without touching the sediments using a cutter or router mounted on an adequate frame (Meisburger et al. 1980). The sectioning is

then completed with a knife. This method generally disturbs the sediments less than extrusion would (ASTM 1997).

- Since this operation requires a number of different tools, subsampling must be done in the laboratory. This means that care should be taken in transporting the samples to maintain the cores upright, minimize vibrations and ensure a storage temperature of roughly 4°C. Plans for shipping containers of ice are given in ASTM (1997).
- Precautions should also be taken in removing cutting debris from sediments with tweezers.
- Once the external liner has been cut, the core is sectioned with fine monofilament line in the case of sand or clay sediments. Clay sediments can also be cut with a knife. For sediments with a particularly high clay content, an electro-osmotic spatula can be used (ASTM 1997, Bouma 1969). Insulated gloves should be worn as protection against electrical shock.
- Once the core has been sectioned and the two parts separated, the description of the core can be completed. This allows the entire sediment column to be seen in a single glance. Depending on the volume of samples required, one section can be preserved for this purpose, but should be properly wrapped to prevent the sediments from drying out.
- A combination method, using horizontal extrusion and longitudinal sectioning, can be used if sediments are compact enough.

Sectioning in defined lengths. If disposable tubes are used, they can also be employed to preserve subsamples. In this case, cores can be cut in sections of a predefined length.

- First, the tube must be marked at regular intervals, beginning at the top, to identify the sections correctly.
- To prevent sediments from oozing out at the cuts, sediments can be frozen first. To do this, the tube is inserted in a box specially designed to hold dry ice (ASTM 1997). The box is five times the diameter of the tube. A 4-cm core will take around 25 minutes to freeze using dry ice. Freezing is not recommended if certain ecotoxicological assays and analyses are being conducted.[6]
- The core must be cut quickly using a hacksaw before it thaws out. The ends can be scraped with a small stainless steel spatula to remove any potential contamination.
- The ends of the sections are then sealed with polyethylene or polystyrene stoppers.

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[6] Sample preservation and handling conditions in the field must be submitted beforehand to the person in charge of the analyses to determine the constraints related to the analyses and bioassays to be carried out.

6.1.3 Recovering samples from grab samplers

- Before removing the samples, the exterior of the sampler must be rinsed thoroughly to prevent cross contamination.
- The bucket should be placed in a container filled with water before taking it out of the water to maintain the hydraulic head.
- Depending on the model, the grab must be put on a stand so that subsampling can be done directly from the bucket or so that the sample can be removed and put into a stainless steel container. The entire working surface must be covered with Teflon®, high density polyethylene or other inert material.
- The sample must be allowed to sit for a few minutes to allow any resuspended particles to settle.
- Overlying water must be carefully siphoned off or decanted to avoid disturbing the fine surface sediments.

a) Subsampling. Subsampling from a grab sampler is carried out in the field only. Subsampling instruments must be made of the compatible material (stainless steel, Teflon® coating, polyethylene, etc.) and properly conditioned, in accordance with the type of analyses planned.

- If the sample acceptability criteria are met, the sample is described (see Section 7.1) and then subsampled.
- Water overlying the sample should be removed after the sample has settled.
- A single grab sample is usually not enough to meet analysis needs. Subsamples are therefore transferred into a homogenization container.
- Ideally, homogenization should be done in a glove box or bag filled with nitrogen or argon gas (denser), particularly if AVS's[7] are to be analysed or bioassays are to be carried out (ASTM 1997, Mudroch and Azcue 1995).
- If homogenization cannot be done under these conditions or AVS's are to be measured, an unhomogenized aliquot of the sediments can be put in a glass container, taking care to fill it to the very top to reduce oxidation and to keep it at 4°C (ASTM 1997). A similar procedure is used to analyse total sulfides and volatile organic compounds.
- All debris, organisms, pebbles and other objects that are not part of the sample to be analysed should be removed, as part of a preliminary cursory cleaning. A more thorough cleaning can be done later in the laboratory using sieving, depending on the type of analysis. If preliminary cleaning is carried out, it must be recorded in the field notebook.

[7] To prevent losses of AVS's through oxidation.

- Foreign objects should be extracted by hand with tweezers or a sieve before homogenization. The purpose is to separate the foreign objects (organisms, twigs, etc.) from the sediments. Sieving should be used when hand sorting is impractical; the former is best done in the laboratory without using water. When analyses of inorganic compounds are planned, a sieve of stainless steel or plastic with openings of the appropriate size is recommended (ASTM 1997).
- If a sieve is used, the treated sediments should be collected in a precleaned stainless steel container (see cleaning procedures described in Chapter 8). When manual sorting is done, the contents of the sample can be emptied directly into the container.
- Sediments directly in contact with the walls of the sampler should not be used.
- Subsampling can be done using an appropriate ladle or spoon.

***b)* Homogenization.** This is a crucial operation since most of the variability in analysis results is due to the steps involved in sediment homogenization and division.

- A container of sufficient volume must be available for the sample or samples (in the case of a composite sample).
- Sediments are then stirred by hand with a spatula (made of inert materials and pre-treated as specified) or mechanically until a homogeneous consistency, texture and colour are obtained. Regardless of the method used, it must be standardized to ensure consistency and minimize the stirring time to limit the risks of altering the particle size distribution of the sediments in the sample (Mudroch and Azcue 1995).
- After homogenization, the sediments must be divided, by the quartering technique, for example. In this method, non-cohesive sediments are moulded into a cone or cake shape and divided into four parts. The opposite quarters are removed and mixed. The divided sample is then mixed, formed again into a cone or cake and divided into four parts as many times as required to obtain the volume desired for subsamples (Environment Canada 1994). In the case of cohesive sediments (clay), it is better to remove subsamples directly from the homogenization container with a spatula, taking care to homogenise continually between samples to remix any sediments that may have settled to the bottom of the pan.

6.2 PORE WATER

If the pore water is to be extracted in the laboratory, sediment samples must be kept at 4°C in the appropriate containers until extraction takes place.

Since most sediments are anoxic, all sampling and subsampling operations must be done in an inert (oxygen-free) environment to prevent oxidization (ASTM 1997).

Once the pore water has been extracted, the following recommendations should be followed to minimize chemical modification of the water.

- The transfer of pore water into appropriate containers must be done in a glove box with an inert atmosphere, under temperatures that reflect the *in situ* temperature.
- If an alternative method is used, it must be validated using similar handling techniques in a controlled atmosphere.
- Routine measurements should be taken of the pore water: pH, hardness, alkalinity, dissolved oxygen, sulfide and ammonia concentrations (Ankley and Thomas 1992).

7 Field Measurements and Observations

Field notes are an essential supplement to the data gathered and assist greatly in the interpretation of the results of the analyses. They are used to validate data quality indicators, identify the exact location of samples and collate field measurements, sampling dates and times and any specific conditions or events that could affect results. Any failure to implement, or modifications in, SOPs must also be documented and explained in the field notes.

The information (essential, desirable or useful elements) to be recorded is shown in detail in Appendix D.1.

7.1 DESCRIPTION OF SAMPLES

The description of samples provides important information on the nature of sediments and allows them to be compared in view of obtaining composite samplings. It is strongly suggested that one person do all the sample descriptions to ensure consistency. Similarly, one person should be assigned to homogenizing samples and putting them in containers, each of which, without exception, must be accompanied by an identification form (Appendix D.2).

Methods for the comprehensive description and identification of sediments (visual and manual) are given in ASTM (1997) and U.S. ACE (1996). For dredging projects, description is limited to the following parameters:

- Particle size distribution

Determine, using Wentworth's classification (Table D.1), the relative proportion (Appendix D, Figure D.1) of the different particle fractions and the overall appearance of the sample (Appendix D, Figure D.2). This applies mainly to coarse sediments (i.e. when the coarse fraction percentage [sand, gravel, pebbles] is over 50%). A comparison device can be easily made using the diagram shown in Figure D.3 of Appendix D. The presence of debris of fossil or plant origin should also be noted and quantified.

- Shape

Coarse sediments (sand, gravel, pebbles) should be described according to particle shape (Appendix D, Figure D.4). The shape of the sediments may provide important information on their origins and mode of deposition.

- Colour

Determine the colour of sediments with the naked eye. This should be done quickly, on a clean surface using a Munsell colour chart (Munsell Color Company and USDA).

- Odour

Identify odours, mainly to determine if hydrocarbons are present. Safety precautions must be taken because of potential health risks.

- Moisture content

Qualify the moisture content using Table D.2 in Appendix D. Marine clays will have a lower moisture content than recent sediments.

- Consistency

Categorize cohesive sediments (> 15% clay + silt) according to their consistency (Appendix D, Table D.3). This is not done with coarse, noncohesive sediments (gravel).

- Structure

Identify, in the case of integral samples, internal structures such as horizons and lamina that may provide information on accumulation conditions (erosion, transport and deposition). This must be done for all core samples and whenever possible for grab samples (Appendix D, Table D.4).

- Descriptive name

Identify sediments for classification purposes and to provide information for use in data interpretation. The emphasis is on distinguishing between recent sediments (current hydrodynamic and sediment regime) and ancient sediments (marine clays, glaciolacustrine sediments and glacial sediments).

- Acid reaction

Carry out an acid reaction test on an aliquot to determine if carbonates are present in the sediments, interpreting the results using the criteria in Table D.5 of Appendix D. The presence of carbonates may be important for the adsorption of some metals.

In the case of core samples, it can also be useful to describe the stratigraphy of sediments using a specific form with a vertical scale (Appendix D, Table D.6).

Lastly, it is recommended that samples be photographed before homogenization. Photographs will provide a record of the external characteristics of the sample before handling and can be used to emphasize an important feature or detail or to validate the description. Cores should also be photographed to show such things as stratigraphic changes and sampling artefacts. The sample number should appear in each photo with a scale indicator. Photos should be numbered and the number recorded on the sample description sheet.

7.2 MEASURING PHYSICOCHEMICAL CHARACTERISTICS

The oxidation-reduction potential (Eh) and pH of sediments should always be measured immediately after the sample is taken (on undisturbed sediments) to reduce the risk that the measured values will be affected by changes in sediment chemistry (Mudroch and Azcue 1995).

If required, an aliquot can be taken simultaneously and kept at 4°C for the subsequent measurement of cation exchange capacity (CEC) in the laboratory (Mudroch and Azcue 1995).

7.2.1 Measurement of Eh

Interpreting Eh measurements in an aquatic environment poses several problems (Brassard 1997):

- Difficulties associated with the disturbance of sediments, resulting in the release or adsorption of gases (particularly O_2 and H_2S) and reactions at the liquid junction of the reference electrode such as sulfide precipitation
- Instability and poor reproducibility of measurements, due to low exchange current densities at the surface of the platinum electrode
- Variable response of platinum electrode depending on environmental characteristics (e.g. the formation of platinum sulfide on the electrode in a reduction environment may generate negative potentials).

These limitations must be taken into account when measuring Eh. Procedures are described in detail in Appendix D.4.

7.2.2 Measurement of pH

Although pH measurements are more reliable than those of Eh, certain problems arise because of the complex interactions between the various sediment components (sulfates, iron and manganese oxides, carbonates and silicates).

Since only slight pH variations are to be expected in the aquatic environments in question — 6.5–7.5 in estuary environments and 6.5–7.3 in marine environments (Brassard 1997) — an instrument with an accuracy of 0.01 is required.

The recommended procedure for measuring pH is described in Appendix D.4.

8 Sample Preservation Methods

8.1 SEDIMENTS

8.1.1 Sample handling and transport

Containers and equipment used for handling sediments should be carefully chosen to prevent sample loss or contamination (Mudroch and Azcue 1995). Samples to be used for trace metal analyses and bioassays must not come into contact with metal surfaces, while samples for organic analyses must not come into contact with plastic surfaces.

In the field, samples can be kept in ice chests, using cooling packs or Nalgene bottles filled with water and frozen. Ice and meltwater should not come into direct contact with sample containers. As an extra precaution, containers can be kept inside plastic bags to keep them dry, which also prevents the loss of the label or cross contamination should a container break during transport. If samples are to be frozen in the field, dry ice can be used. When samples are to be frozen, containers should not be filled to the top, to allow for expansion. Samples meant for toxicity assays must not be frozen.

It is also important to use a reliable shipper to ensure samples arrive at the laboratory within the deadlines required for analyses.

8.1.2 Preservation of samples

Tables E.1 and E.2 in Appendix E show the preservation techniques usually required during a sampling campaign, including the quantity of samples required, type of container and maximum time between collection and physicochemical analysis or bioassays. Preservatives are not to be added to sediment samples (Walton 1980).

These methods are closely tied to the analytical methods used since detection and quantification limits may also determine sample quantity and type. In addition, analysis methods may affect the choice of containers and sample preservation techniques.

In general, plastic, glass and Teflon® containers are used for the analysis of inorganic contaminants, while glass is used for organic contaminants. Volatile compounds require that containers be filled to their capacity. It is crucial to work in co-operation with laboratory personnel to obtain the additional information required.

8.1.3 Cleaning of containers

It is essential to ensure that containers and stoppers used for sampling and/or sample preservation, if they are not the same, do not cause contamination. Generally, laboratory personnel are responsible for preparing and cleaning containers. The main steps in preparing and cleaning containers and stoppers are outlined below:

- Usually, containers are washed with a phosphate-free detergent by scrubbing and then rinsing all surfaces under the tap, followed by a thorough rinse with deionized or distilled water.
- When analyses of inorganic chemicals are to be carried out, containers must also be soaked in a 10% nitric acid solution and then rinsed thoroughly with deionized or distilled water.
- In the case of analyses of organic chemicals, containers must usually be rinsed with various organic solvents (e.g. acetone, hexane), followed by a thorough rinse with deionized or distilled water. Table E.3 in Appendix E gives additional information on the subject.

8.1.4 Cleaning of sampling equipment

The same procedures described for cleaning containers are used for decontaminating equipment and materials that come in contact with samples. Table E.3 in Appendix E summarizes the procedures involved. An alternate method is given in ASTM (1997), which involves conditioning new plastic containers for seven days in HCl, HNO3 and deionized water solutions.

8.2 PORE WATER

8.2.1 Sample handling and transport

The same basic precautions described in 8.1.1 are required for pore water samples.

8.2.2 Preservation of samples

Table E.4 in Appendix E shows the preservation techniques usually required in a pore water sampling campaign. It defines in general the quantity of samples required, type of containers used and maximum time between collection and analysis. Ideally, samples should be stored for no more than two weeks in an anoxic environment at a storage temperature of

roughly 4°C. Preservatives are sometimes added to pore water samples, unlike sediment samples (Environment Canada 1994).

In addition, plastic containers must be stored in an anoxic environment before sampling to prevent the release of oxygen adsorbed in the walls.

These methods are closely tied to the analytical methods used since detection and quantification limits may also determine sample quantity and type. In addition, analysis methods may affect the choice of containers and sample preservation techniques, as described in Section 8.1.2.

8.2.3 Cleaning of containers

The same procedures described in Section 8.1.3 are used to clean containers for pore water samples. Table E.5 in Appendix E provides additional information on pore water samples.

8.2.4 Cleaning of sampling equipment

The same procedures described for cleaning containers are used for decontaminating the equipment and materials that come in contact with pore water samples. Table E.5 in Appendix E summarizes the procedures required.

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Appendix A Corers

A.1 SHAPE PARAMETERS OF CORERS

Cutting angle

The cutting angle of the cutting head, or core tube if no head is used, must be less than 10°, with the optimum angle being 5° (Figure A.1). An angle of 2° works well but the edge of the head is easily damaged. The cutting angle must be decreased if the surface ratio (see below) is over 5% (U.S. ACE 1996).

Diameter of corer

Increasing the diameter allows a decrease in both internal friction and the relative significance of the washout of outer sediments as a function of the volume sampled (Blomqvist 1985). With a diameter of greater than 8 cm, however, the extent of core reduction caused by friction decreases asymptotically. If the diameter is smaller, friction increases linearly and even exponentially (Figure A.2). Moreover, sediment type and penetration speed and method also influence core reduction. Given the relative importance of these factors, reduction is not linear and even varies for the same corer, making profile correction and standardization impossible. The minimum internal diameter for corers is 5 cm (ASTM 1997), but a diameter of 10 cm appears to be a good compromise (ASTM 1997). The best performance was found with a 15-cm corer (Blomqvist 1985, Parker and Sills 1990).

Surface ratio

The surface ratio (C_a) is probably the most important factor (Hvorslev 1949) affecting sample disturbance (Figure A.3). This ratio is used to assess the volume of sediments displaced in relation to the volume sampled. The surface ratio is obtained with the following formula:

$$C_a = \frac{\left(D_w^2 - D_c^2\right)}{D_c^2}$$

$C_a < 0.10$ to 0.15

A greater surface ratio can be used if the cutting angle of the head is decreased.

Internal friction ratio

The friction of sediments against the internal walls of the corer may be decreased by increasing the internal diameter of the liner in relation to the internal diameter of the cutting head. This increase allows the sediments to slide more easily. The internal friction ratio is obtained with the following formula:

$$C_i = \frac{D_i - D_e}{D_e}$$

A ratio of 0–0.01 is acceptable for short cores (< 0.5 m), while one of 0.005–0.03 is acceptable for cores of medium length (6–8 times the diameter) and a larger ratio is allowable for longer corers. In most cases, and for medium-length cores (6–8 times the diameter) up to 2 m, the ratio should be 0.0075 to 0.015 (ASTM 1997).

If the tube is equipped with a flexible liner with internal distributor (Figure A.3), as the Swedish foil sampler and Delft stocking sampler are (U.S. ACE 1996), internal friction is reduced so effectively that a smaller surface ratio can be used ($C_a < 0.005$). A flexible liner can also be used with other coring techniques.

The resulting decrease in recovery can be described by the recovery ratio ($r = L/l$) where L is the corer penetration depth and l, the length of the core (without washout during recovery). This ratio must be determined for each core when sampling.

It is crucial to understand that reduced penetration results in the inadequate sampling of some strata or levels (Blomqvist 1985, Blomqvist 1990, Lebel et al. 1982) rather than sediment compaction (Figure A.2). The mechanics of internal friction are complex (Figure A.2) and vary depending on the corer diameter (Blomqvist 1985), penetration speed (Figure A.2) and nature of the sediments. Although the decrease in recovery is greater in silty and clay sediments (Leonard 1990) than in very porous organic sediments (Blomqvist 1991), it may vary from site to site (Leonard 1990). The stratification of sediments into horizons of different composition and compactness also reduces penetration, which will vary during the penetration of the corer (Blomqvist 1990, Hvorslev 1949, Hongve and Erlandsen 1979).

High penetration speed is another element that promotes reduced penetration. In fact, it is strongly recommended that the corer be allowed to penetrate the sediments by its own weight (Blomqvist 1990). Using supplementary ballast compensates, up to a certain

point, for poor penetration, and the use of a piston also improves penetration (U.S. ACE 1996, ASTM 1997). Some corers have been modified so that they can penetrate more deeply by percussion using mobile ballast. If required, penetration speed can be controlled and kept below 1 m/sec; in the case of freefall, the height should ideally be 1 m, or at the most, under 3 m. A penetration speed of 2 cm/sec (Chant and Cornett 1991) has even been suggested, which is only feasible if the corer is inserted by hand.

External friction ratio

The external friction in the core tube also affects penetration. This ratio is obtained with the formula:

$$C_o = \frac{D_w - D_t}{D_t}$$

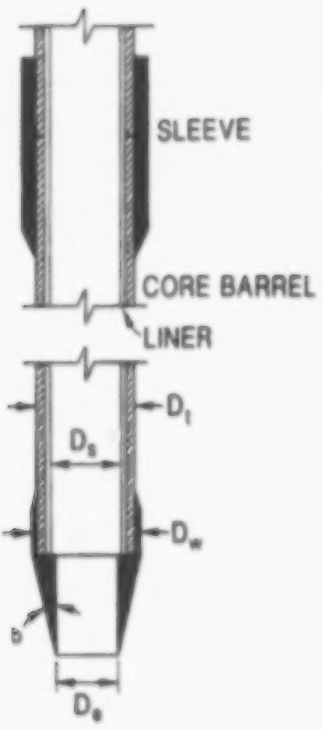
C_o is 0 for noncohesive sediments and between 1 and 3 for cohesive sediments. The greater width of the cutting head in relation to the tube compresses exterior sediments and facilitates corer penetration. For long cores, a supplementary sleeve can be added (Figure A.1).

Length of corer tube

The length of the corer tube is a function of the diameter and must be slightly greater than the greatest depth (thickness) of sediment that can be sampled without causing significant sediment disturbance (L). The corer length factor (L_f) is obtained with the formula:

$$L_f = \frac{L}{D_t}$$

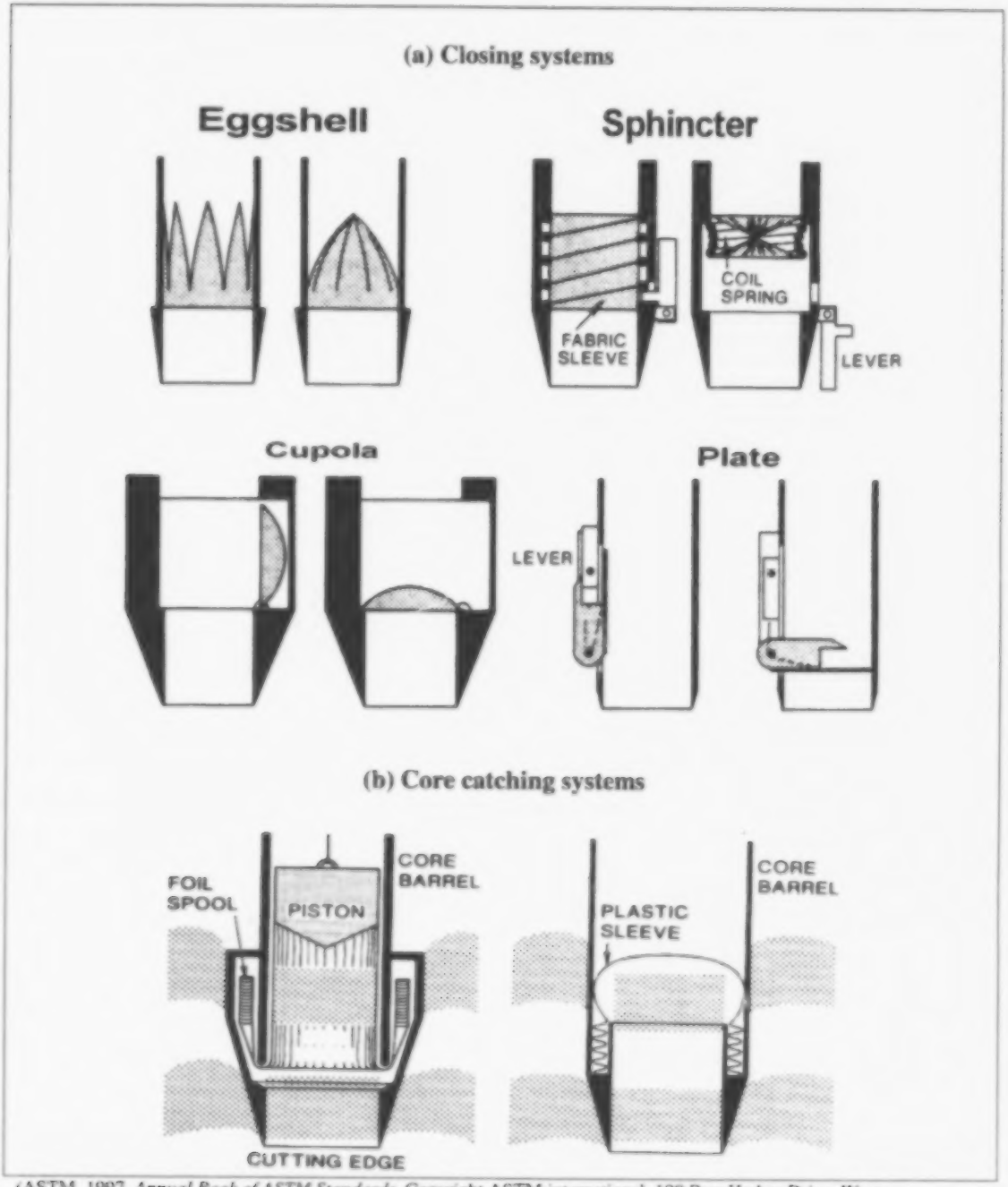
L_f must be less than 5–10 in the case of cohesive sediments and 10–20 for noncohesive sediments. The tube diameter must therefore be selected based on the sediment type. The factor can be increased by using a piston or increasing penetration speed, taking into account disturbance factors intrinsic to an increase in penetration speed.



b cutting angle of cutting head
de internal diameter of cutting head
ds internal diameter of corer tube (or liner when present)
dw external diameter of cutting head
dt external diameter of corer tube

(ASTM, 1997, *Annual Book of ASTM Standards*, Copyright ASTM international, 100 Barr Harbor Drive, West Conshohocken, PA 19428, p. 284, with permission)

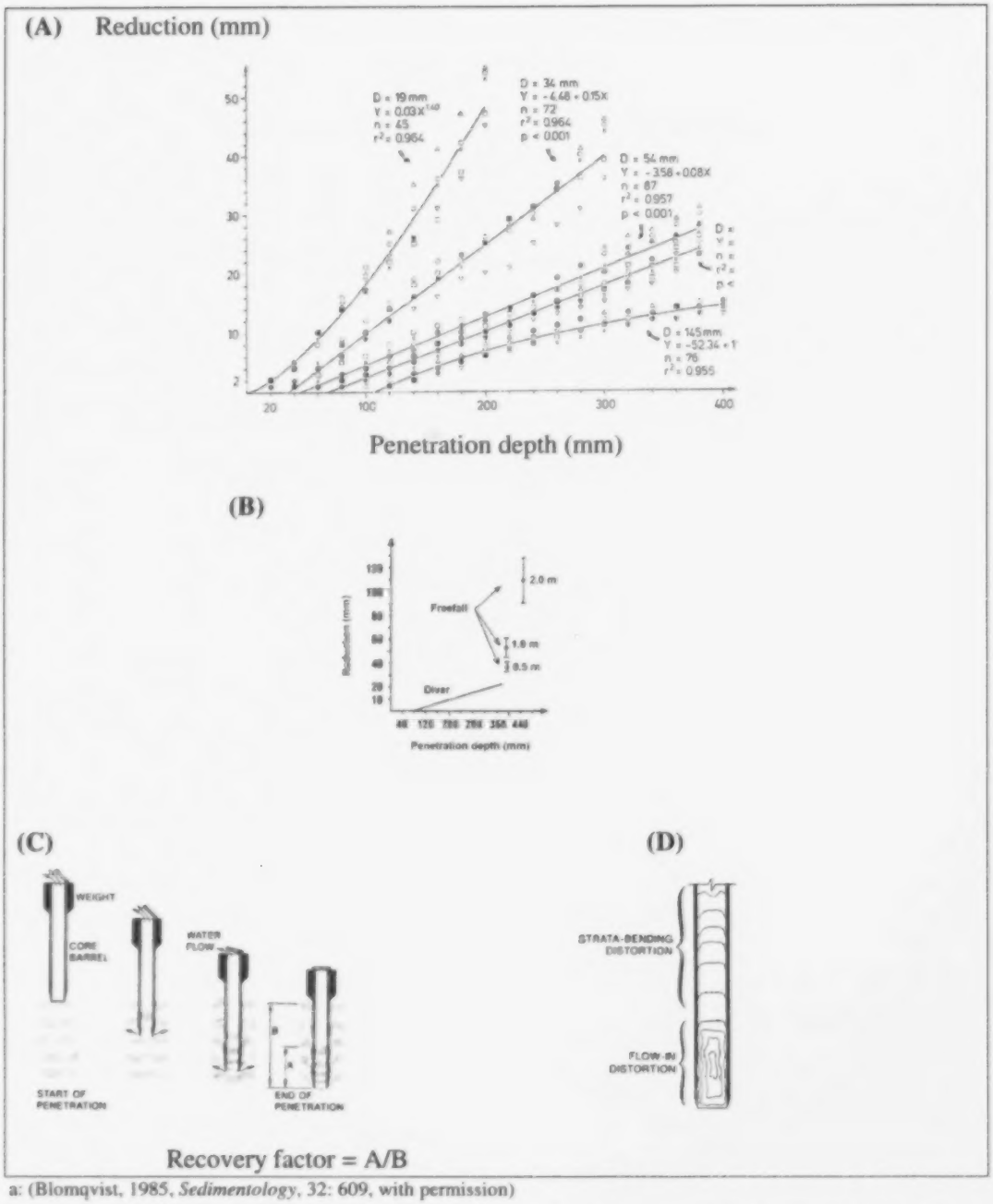
Figure A.1 Major shape parameters for open-barrel and piston corers



(ASTM, 1997, *Annual Book of ASTM Standards*, Copyright ASTM international, 100 Barr Harbor Drive, West Conshohocken, PA 19428, p. 283, with permission).

Note: Some mechanisms disturb sediments during penetration (eggshell core catcher) while others can only be used with large-diameter core tubes (i.e. sphincter) to respect Hvorslev's parameters.

Figure A.2 Types of corer closing mechanisms (a) and core catchers (b) used to (b) Core catching systems reduce internal friction

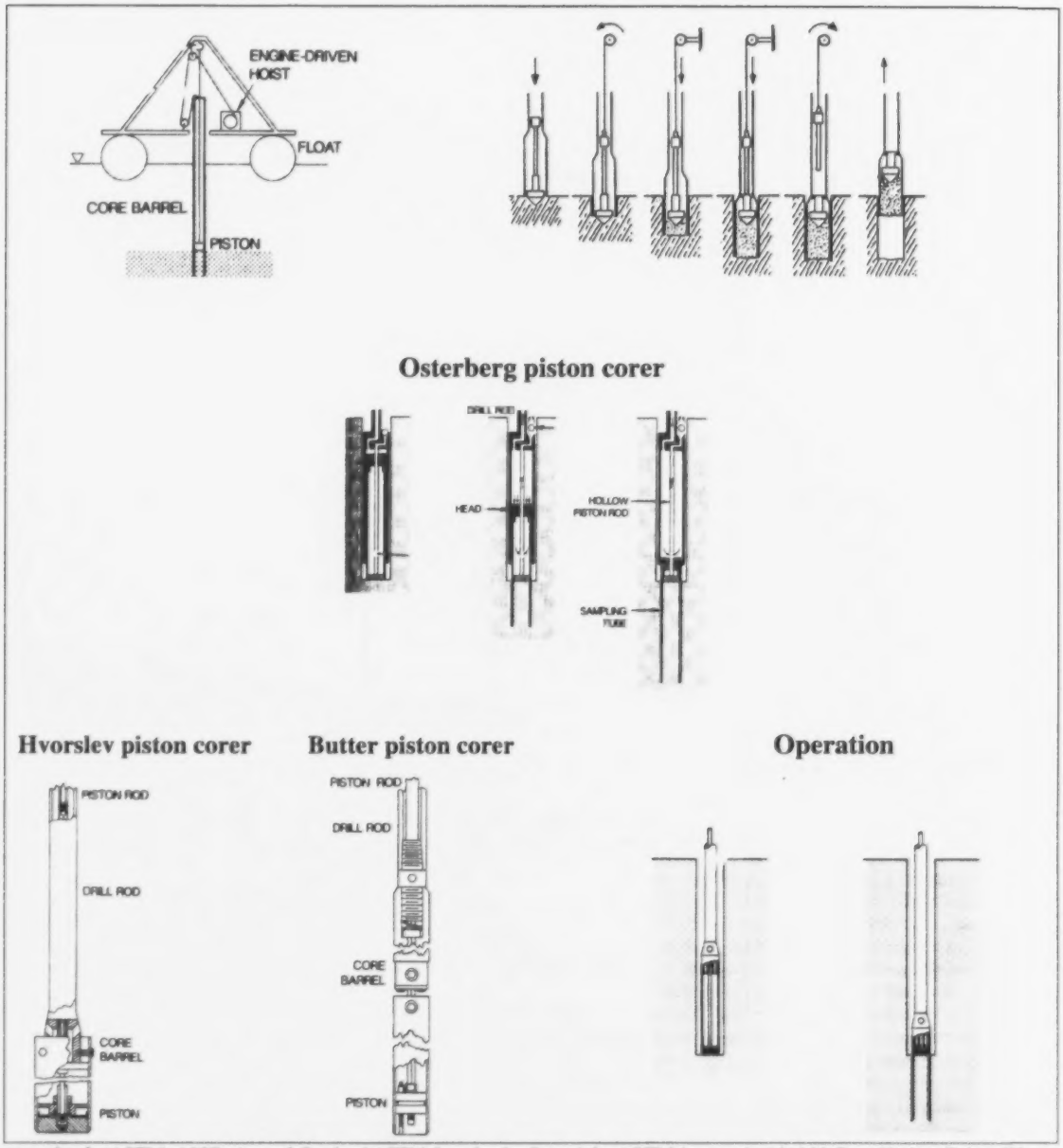


a: (Blomqvist, 1985, *Sedimentology*, 32: 609, with permission)

c et d : (ASTM, 1997, *Annual Book of ASTM Standards*, Copyright ASTM international, 100 Barr Harbor Drive, West Conshohocken, PA 19428, p. 283-284, with permission)

Figure A.3 Importance of reduction of core size (a) as a function of core tube diameter (b) and penetration speed (c). Sampling artefacts caused by internal friction in the tube (washout) and stopper suction due to a poorly adjusted piston (d)

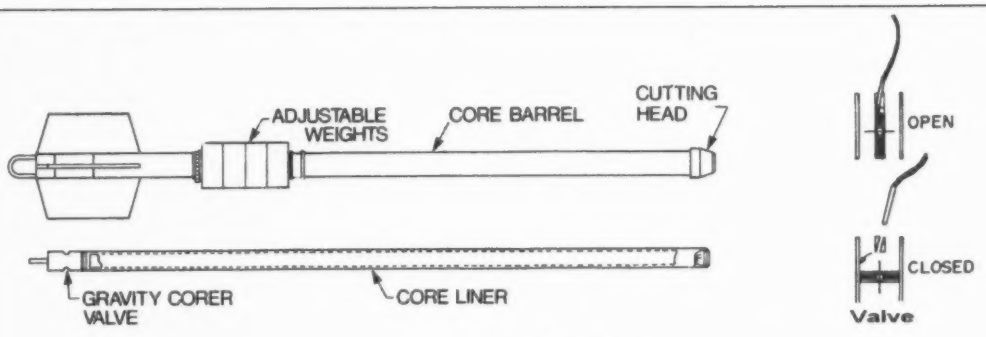
A.2 TYPES OF CORERS



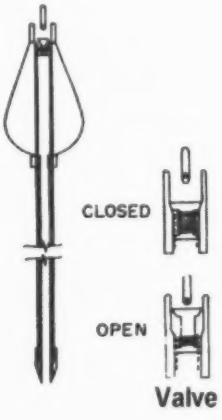
a-c-d-e-f: (ASTM, 1997, *Annual Book of ASTM Standards*, Copyright ASTM international, 100 Barr Harbor Drive, West Conshohocken, PA 19428, p. 288-289, with permission)

b: (Robbe, 1981, *Pollutions métalliques du milieu naturel: Guide méthodologique de leur étude à partir des sédiments*, with permission)

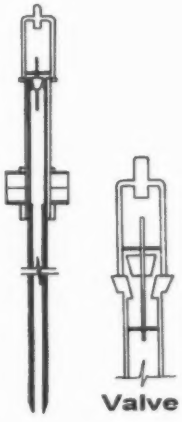
Figure A.4 Design and operating principles of piston corers



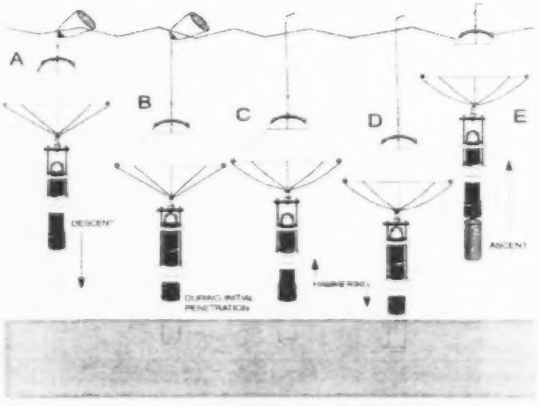
Benthos



Alpine



Phleger



Algonquin

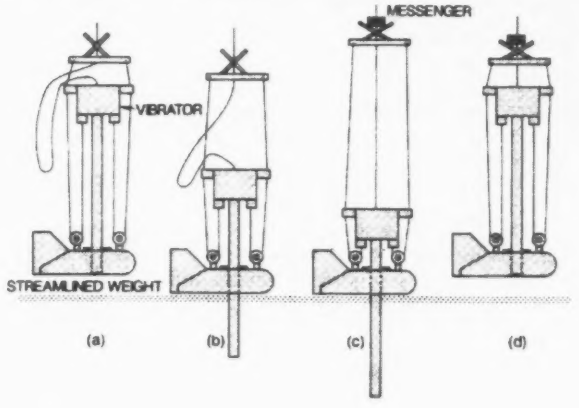
(Cushing *et al.*, 1997, *Journal of Paleolimnology*, 18 :309, with permission of Kluwer Academic Publishers)

(Mudroch et MacKnight, 1994, *Techniques for aquatic sediments sampling*, p.42 et 46, with permission)

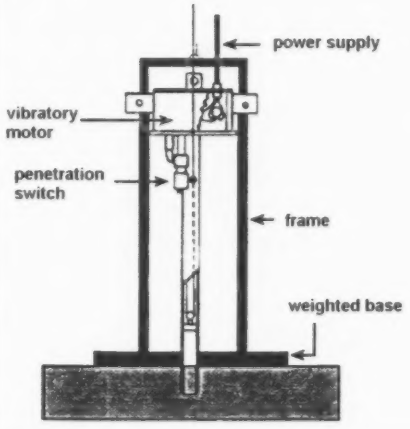
Figure A.5 Design and operating principles of open-barrel corers

Prych-Hubbell

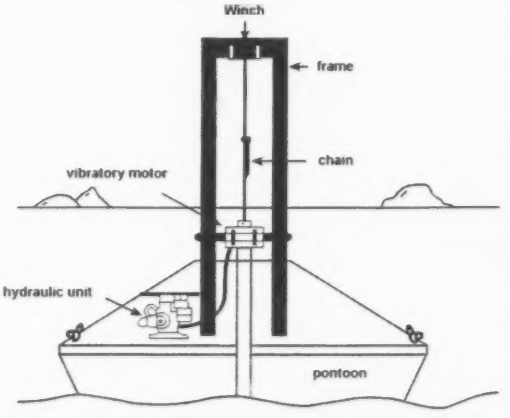
(ASTM, 1997, *Annual Book of ASTM Standards*, Copyright ASTM international, 100 Barr Harbor Drive, West Conshohocken, PA 19428, p. 290, with permission)



Submergible (e.g. Rossfelder)



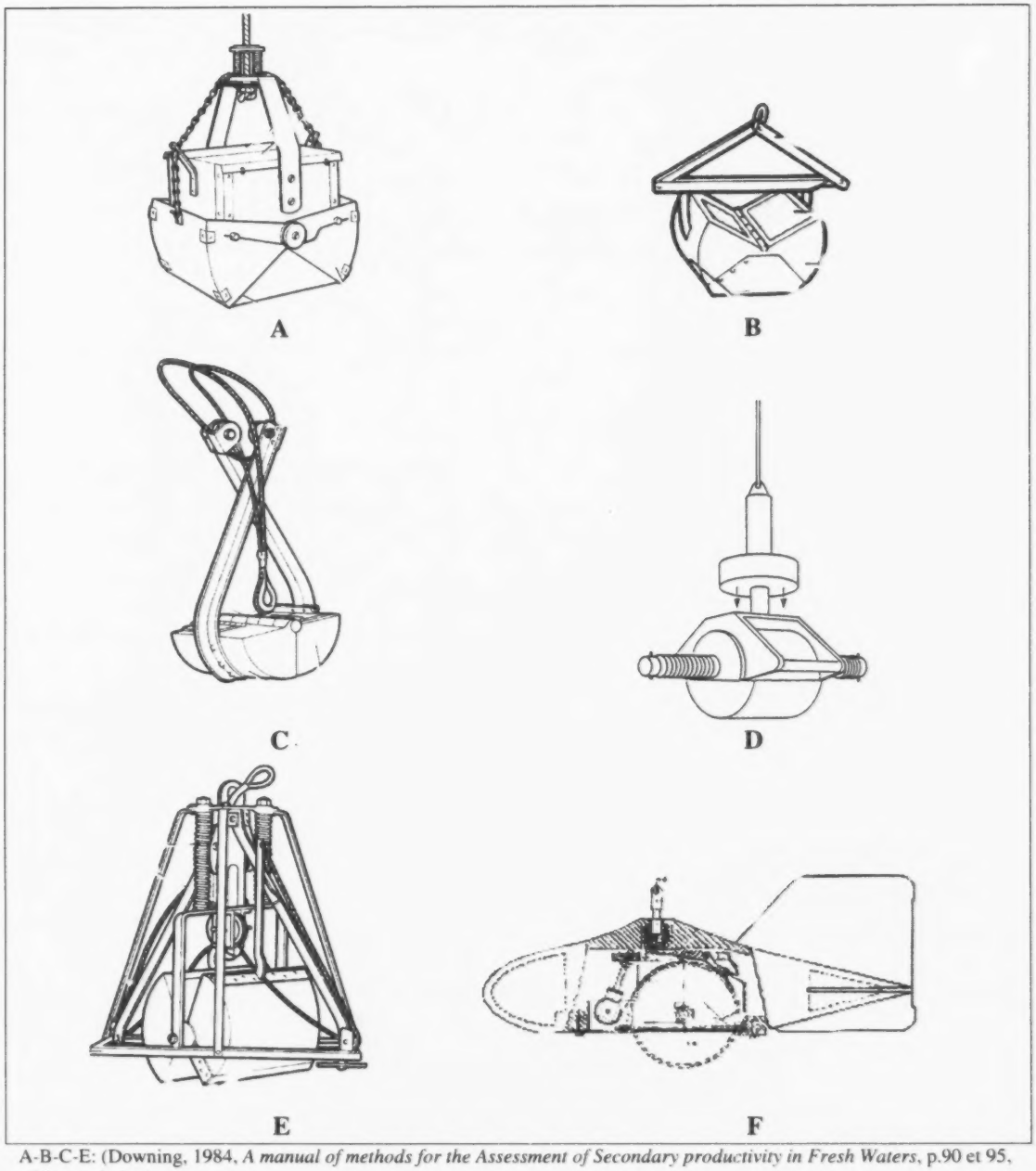
Surface-operated vibratory corer (e.g. Meta-drill)



(Modified from USACE, 1996)

Figure A.6 Design and operating principles of vibratory corers

Appendix B Grab Samplers



A-B-C-E: (Downing, 1984, *A manual of methods for the Assessment of Secondary productivity in Fresh Waters*, p.90 et 95, with permission)
D: (Mudroch et Azcue, 1995, *Manual of aquatic sediment sampling*, p.58, with permission)
F: (Rickly hydrological Co, 2002, *Bed-material samplers, US-BMH-60 &US BM-54*, with permission)

Figure B.1 Recommended grab samplers: (a) Ekman; (b) Ponar; (c) Van Veen; (d) Shipek; (e) Smith-McIntyre; (f) US BM-54

Appendix C General Field Procedures

Table C.1
Checklist for preparing standard operating procedures (SOPs)

What observations must be made and recorded at the sampling site?
Has information on DQOs, analysis methods, SDDs, etc. been included?
Have specific instructions on modifying protocols in case of a problem been provided?
Has a list of all sampling equipment been prepared?
- Does this list include all sampling devices?
- Are all sample containers included?
- Is container composition appropriate for substances to be tested?
- Does container size correspond to sample amount required?
- Are all chemical preservatives and preservation equipment noted?
- Are materials for cleaning equipment included?
- Are labels, gummed tape, permanent ink pens and packaging on the list?
- Are chain of custody forms and seals for samples included?
- Are chemical-resistant clothing and other safety equipment included?
Have instructions on how to fill out sample labels been included?
- Have instructions on equipment calibration and operation been provided?
- Have instructions on cleaning and handling sample containers been provided?
Have instructions been provided for each type of sample?
- Are numbers and size provided for each sample type?
- Do sampling duration or other conditions need to be specified?
- Are the number, type and size of all QC samples indicated?
- Are the number, type and size of exploratory and supplementary samples indicated?
- Are instructions on preparing composite samples needed?
- Are instructions on field preparations and measurements provided?
- Are instructions for disposing of sample waste included?
Are instructions on filling out sample labels included?
- Is the maximum shelf life for samples indicated?
Have the project timetable and responsibility line been determined?
Have instructions for packaging, shipping and storage been provided?
Have instructions on the chain of custody been included?
Have safety plans been included?

(Modified from CCME, 1993)

Appendix D Description of Samples

.1 FIELD MEASUREMENTS AND OBSERVATIONS

▪ Identification of project
▪ Station number and identification of cores
▪ Date and time of sampling, date and time of collection of each sample (including replicates) and handling of subsamples
▪ Location (longitude, latitude, datum)
▪ Water level
▪ Weather conditions, waves, currents, etc.
▪ Type of vessel used
▪ Type of sampler (grab, corer)
▪ Corer characteristics (length, characteristic factors [see below])
▪ Height of freefall (grabs and corers)
▪ Total weight of sampler, weight of ballast, use of percussion
▪ Sampler penetration, depth of subsampling intervals
▪ Total core length, length of subsamples
▪ Field measurements, including (where applicable) pH, Eh, acid reaction test (carbonates), water/sediment temperatures, dissolved oxygen (water) and conductivity/salinity (estuary and marine environments)
▪ Description of sediments: - nature of sediments (Table D.1) - texture of sediments (Tables D.2, D.3) - structure of sediments (Table D.4) - presence/absence of carbonates (Table D.5) - odour - colour (Munsell colour chart) - presence/absence of invertebrates, debris
▪ Orientation of cores during storage (vertical, horizontal)
▪ Storage temperature
▪ Members of field team
▪ Person in charge of description, subsampling

D.2 INFORMATION FORM: REQUIRED SAMPLING DATA

(One form to be filled out per sampling station)

Date and time:

Identification and location of sampling station:

Geographic co-ordinates:

Description of station:
(including landmarks, weather conditions, dominant currents and winds, position in intertidal zone if station not in sublittoral zone, water depth, etc.)

Information on sample:

Estimated volume and weight of sample:

Number of passes required for grab or corer to take sample:

Estimated drift between samples:
(to give idea of area in question, take account of depth, length of mooring lines, winds and currents)

Describe appearance of each sample:
- Photo of sample with ruler and identification tag
- Particle size distribution: tactile, Wentworth scale
- Odour (H_2S, hydrocarbons or other)
- Consistency: fluid, soft, compact, consolidated, noncohesive, etc.
- Colour (Munsell colour chart)
- Presence of organic material, benthic organisms or detritus
- Presence of hydrocarbons (spots on instruments and iridescent sheen), coal dust, ash
- Identification of sample
- Presence of carbonates (tested with 10% hydrochloric acid)
- Description of strata (thickness, structure, colour)

Equipment used:
- Sampler (e.g. Ponar, box corer, etc.)
- Type, capacity and number of containers and samples
- Other equipment in contact with sample
- Type of vessel used and number of anchors, if applicable
- Other equipment used (e.g. winch, echosounder)

Sampling method:
(Describe sampling methods used, including penetration depth and any precautions taken for quality control and assurance purposes)

Labelling, storage and shipping of sampling:
(Describe labelling, storage and shipping operations and conditions under which they took place)

_____ _____

Signature Date

(Modified from Environment Canada, 1994)

D.3 DESCRIPTION OF SAMPLES

TableD.1
Particle size distribution of sediments

Class	Criterion	Size	Identification
Coarse	Blocks	600 mm +	Visible to the naked eye, shape
	Stones	250-600 mm	
	Pebbles	75-250 mm	
	Gravel	2-75 mm	
	Very coarse sand	1-2 mm	Visible to the naked eye
	Coarse sand	0.5-1 mm	
	Medium sand	0.25-0.5 mm	
	Fine sand	0.125-0.25 mm	
	Very fine sand	0.063-0.125	
Fine	Silt	< 63 m	Tactile (granular but invisible to the naked eye) + Tactile (smooth, slimy)
	Clay	< 4 µm	
Organic	Mud		Decomposing plant material

(Modified from Wentworth, 1992 and U.S. ACE, 1996)

Table D.2
Classification by water content

Description	Criterion
Moist	No visible water
Wet	Free water

(ASTM, 1997, *Annual Book of ASTM Standards*, Copyright ASTM international, 100 Barr Harbor Drive, West Conshohocken, PA 19428, p. 290, with permission)

Table D.3
Classification by consistency

Description	Criterion
Fluid	Liquid, no plasticity
Very soft	Penetrated very easily when pressed with the thumb; cohesive, viscous, not firm
Soft	When pressed with the thumb, thumb penetrates over 25 mm; cohesive, plastic but firm
Compact	Cohesive, firm and brittle

(ASTM, 1997, *Annual Book of ASTM Standards*, Copyright ASTM international, 100 Barr Harbor Drive, West Conshohocken, PA 19428, p. 290, with permission)

Table D.4
Descriptive criteria of sediment structure

Description	Criterion	Remarks
Stratified	Alternating layers more then 6 mm thick	Difference in composition or colour
Laminated	Alternating layers less than 6 mm thick	Difference in composition or colour
Fissured	Breaks along defined planes	
Intrusions	Pockets of different material within sediment matrix	Intrusions of sand or clay
Reworked	Mottled look	Without distinct strata or lamina
Nodular	Presence of round compact clay nodules	Nodules can be mistaken for gravel but are brittle and friable
Burrowing	Shallow circular openings on the surface (and deep inside sediments in cores)	Evacuation of biogenic gases
Homogeneous	Similar colour and texture throughout the sediment	

(ASTM, 1997, *Annual Book of ASTM Standards*, Copyright ASTM international, 100 Barr Harbor Drive, West Conshohocken, PA 19428, p. 290, with permission)

Table D.5
Descriptive criteria of HCl acid reaction test

Description	Criterion
None	No visible reaction
Low	Slow, light boil
High	Violent reaction, immediate boil

(ASTM, 1997, *Annual Book of ASTM Standards*, Copyright ASTM international, 100 Barr Harbor Drive, West Conshohocken, PA 19428, p. 290, with permission)

Table D.6
Sample core logging sheet

Core logging sheet

Station:_____ Location:_____

Core:_____ of_____ Project no.:_____

Collection date: _____ Time:_____ Geographic co-ordinates X: _____

Person in charge _____ Datum Y:_____

Penetration (cm) :_____ Depth/elevation (bottom) :_____

Length:_____ Reference (map/photo):_____

Weather conditions:_____

_____

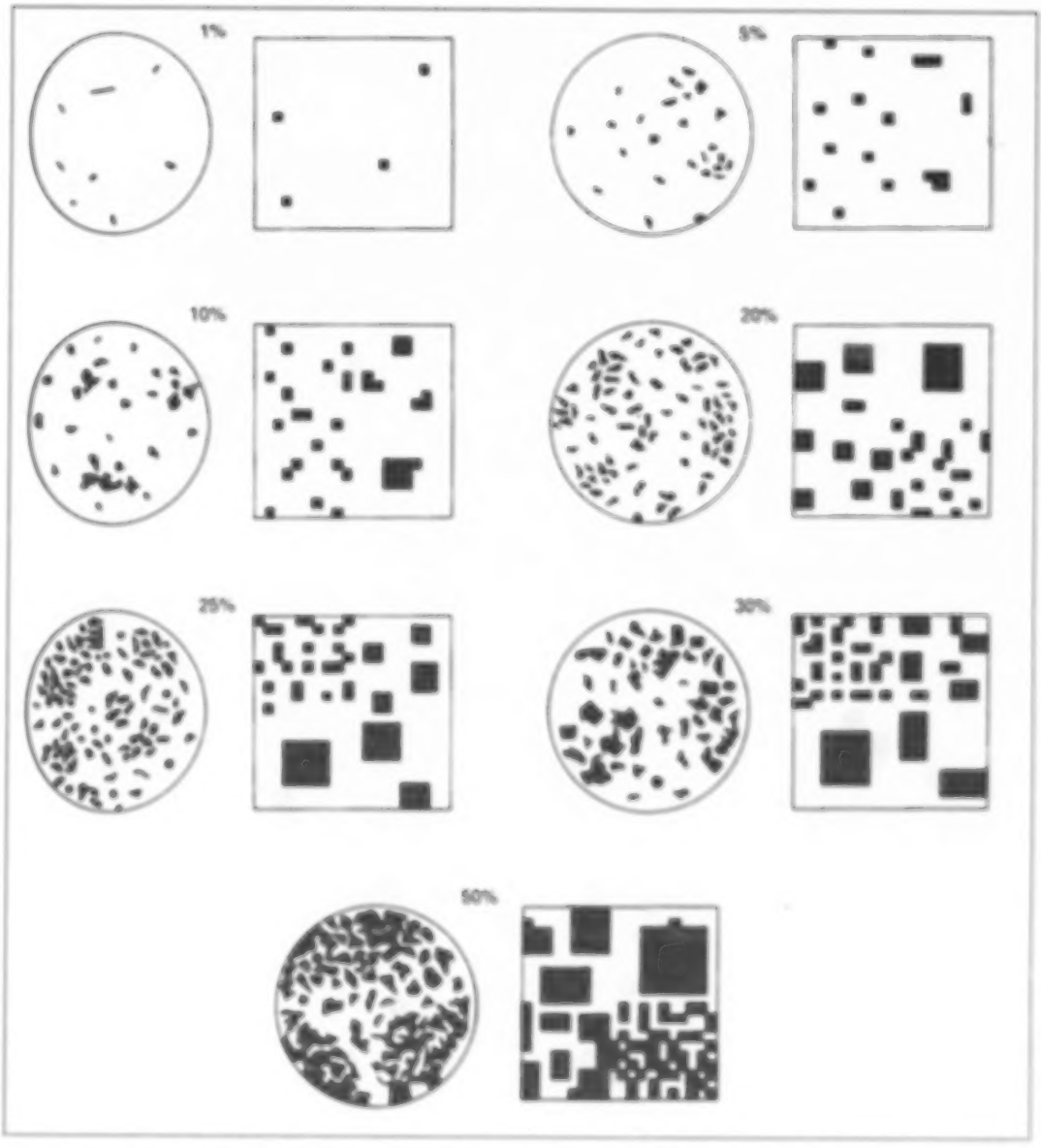
Type of sampling: Hand-held core ☐ 10cm ☐ 12cm Other: ☐ Vibro-corer:____/_____cm

Sampling procedures:_____

_____

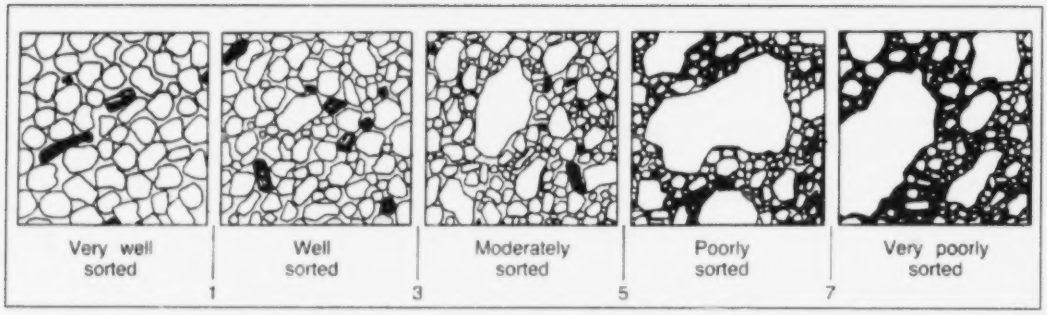
Description						Sample			Comments
Depth (cm)	Lithology	Structure	Biogenic traces Fragments	Odours/traces of contamination	Photo (film, no.)	No.	Interval	Date/time	(temperature, Eh/pH, volume, homogenate, HCl test, other)

Page___of___



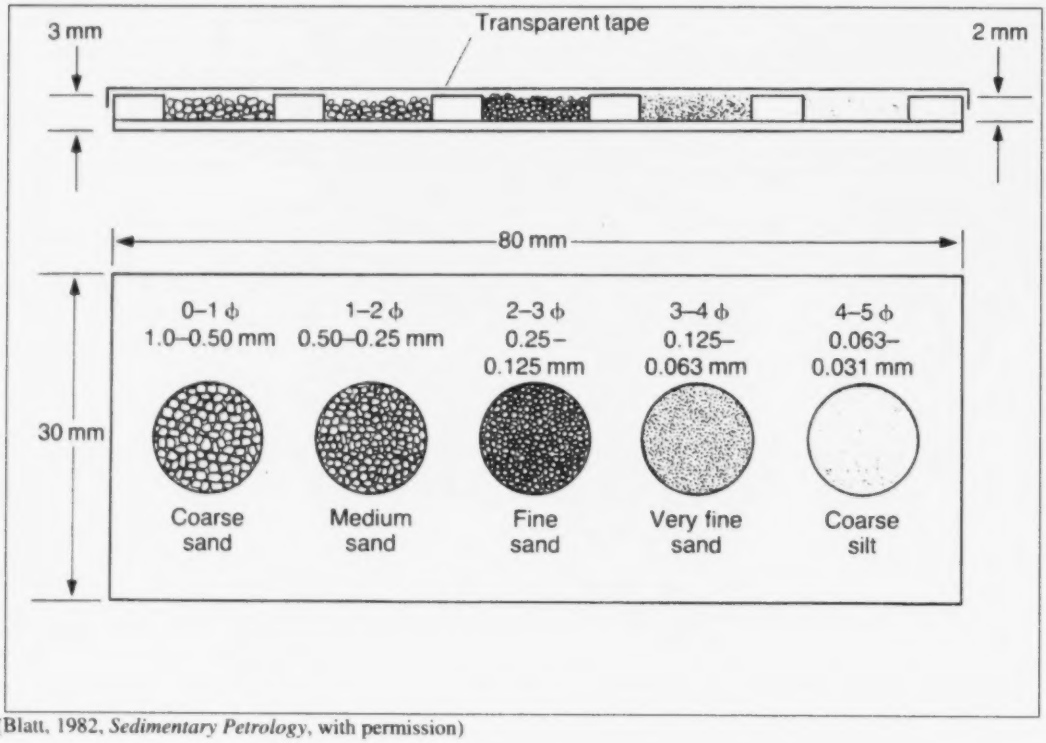
(Folk *et al.*, 1970, *New Zealand Journal of Geology and Geophysics*, 13: 946, with permission)

Figure D.1 Comparative chart of cover percentages



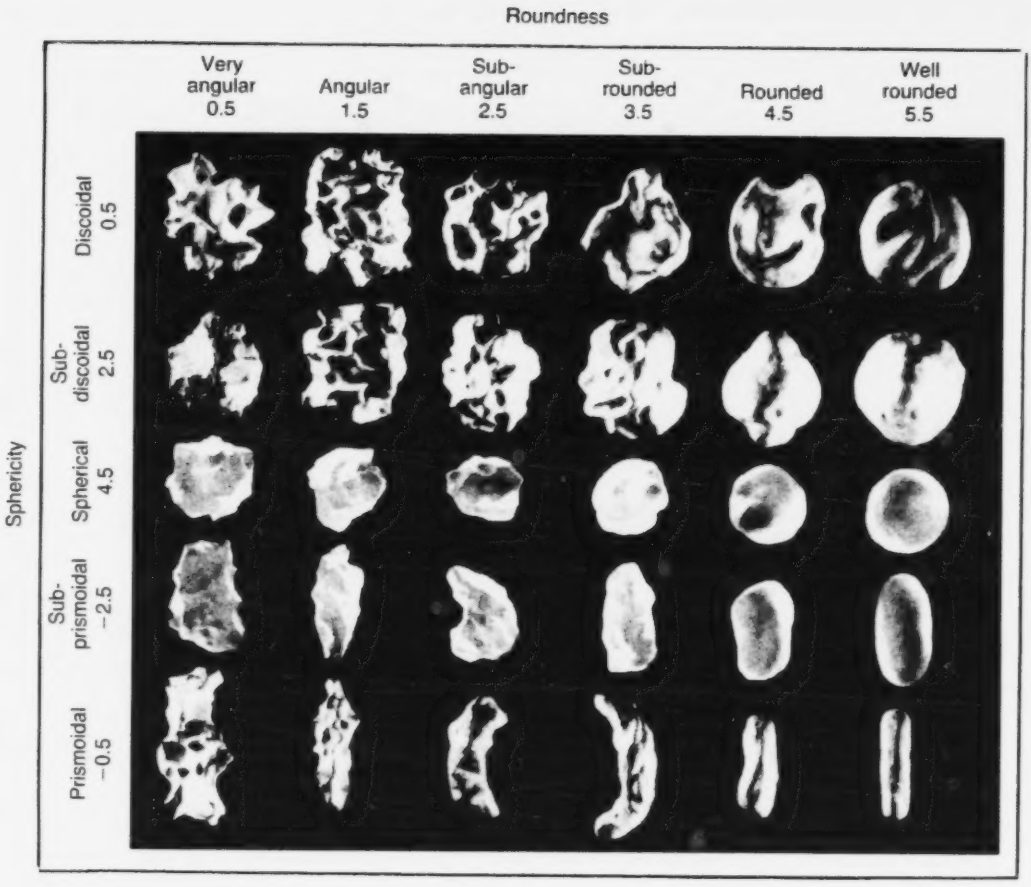
(Modified from Compton, 1962)

Figure D.2 Qualitative assessment of sorting index



(Blatt, 1982, *Sedimentary Petrology*, with permission)

Figure D.3 Particle size analysis comparison tool



(Powers, 1982, *AGI Data sheet 18*, with permission)

Figure D.4 Shape and roundness of coarse particles

D.4 MEASURING pH AND Eh IN THE FIELD

Required equipment

- Portable battery-operated pH/Eh meter (batteries should be recharged if required)
- Combination glass and platinum electrodes or other electrodes suitable for the measurements. Electrodes must be marked at regular intervals to control depth of penetration in sediments.
- Spare electrodes
- Containers for storing electrodes
- Ph buffer solutions (pH 4 and 7)
- Solution for calibration of Eh electrodes such as a Zobell solution (labelled and handled according to safety regulations for cyanides)
- Saturated potassium chloride solution for storage of the electrodes in a plastic bottle
- Other solutions required for the proper functioning of the electrodes as outlined by electrode manufacturer
- Distilled water and tissues for cleaning electrodes
- Support stands and containers for buffer solutions

Measurements

- Allocate a space with all the equipment required for the measurements.
- Electrodes must be connected, checked, calibrated, washed, prepared and acclimatized in station water (when samples are taken from freshwater bodies) before sampling sediments. Eh electrodes must be calibrated with Zobell solution, and pH electrodes with buffer solutions (pH 4 and 7) in a nitrogen atmosphere.
- The sample must remain steady during measurements.
- Electrodes must be inserted carefully in the sediments to the depth marked to avoid any air contamination, particularly around the Eh electrode. Care should be taken not to generate any air pockets between the electrode and the sediments. The pH/Eh meter is switched to the pH scale and the value recorded within one minute after inserting the electrodes.
- Insert the Eh electrode into the sediments to the depth marked and after the potential becomes stabilized, usually after 10-15 minutes, take the Eh measurement, using the mV scale.
- Remove the electrodes from the sediments, rinse them with distilled water and dry with a tissue.
- Electrodes should be calibrated after every fifth measurement, less so for sediment cores.

(Modified from Brassard, 1997)

* Please note that the measurement of temperature is crucial since temperature can have an effect on pH.

Appendix E Preservation of Samples

Table E.1
Suggested volumes and preservation methods for physicochemical analyses of sediment samples[a]

Parameters	Recommended volume (mL)	Type of container	Maximum storage time at 4 ± 2°C[b]
Physical parameters			
PH	100	P, T or G	1 month[c]
Cation exchange capacity	100	T or G	1 month
Moisture	100	P, T or G	1 month
Particle size analysis	400	P, T or G	6 months
Volatile solids	100	P, T or G	1 month
Inorganic parameters			
Total Kjeldahl nitrogen	100	P, T or G	6 months
Total phosphorus	100	P, T or G	6 months
Total mercury	100	P, T or G	1 month
Arsenic, cadmium, chromium, copper, nickel, lead and zinc	100	P, T or G	6 months
Total cyanides	100	P, T or G	6 months
Organic parameters			
Total organic carbon	100	T or G	1 month
Polychlorinated biphenyls	300	GA	14 days
Organochlorine pesticides	300	GA	14 days
Polycyclic aromatic hydrocarbons	300	GB	14 days
Petroleum hydrocarbons C_{10}-C_{50}	300	GA	14 days
Chlorinated dioxins and furans	300	GB	14 days
Phenolic compounds	300	BA	14 days

* This information is taken from the *Guide méthodologique de caractérisation des sédiments*, October 2001 draft version, published by the Centre d'Expertise en Analyse Environnementale du Québec.

Note:

a- No preservative is required for any of the parameters in this table.

b- Samples intended for chemical analyses can be preserved for a longer time (within the maximum time recommended here) by freezing at –15 °C or less. It is important to use glass containers that can withstand freezing to avoid breakage.

c- Ideally, pH should be measured in the field immediately after taking a sample or as soon as possible upon its arrival at the laboratory.

Legend	
TYPE OF CONTAINER	
P	Bottles and stopper linings composed of one of the following plastics: high- or low-density polyethylene, polypropylene, polystyrene, PVC or Teflon®.
T	Bottles and stopper linings composed of the following types of Teflon®: polytetrafluoroethylene (PTFE), fluorinated ethylene-propylene resin (FEP), perfluoroalkoxy (PFA), chlorotrifluoroethylene (CTFE), modified ethylene-tetrafluoroethylene copolymer (ETFE) or ethylene chlorotrifluoroethylene copolymer (ECTFE).
G	Bottles and stopper linings are made of glass, plastic or Teflon®
GA	Bottles are made of glass and stopper linings are made of aluminum (aluminum foil between sample and stopper) or Teflon®
GB	Bottles are made of amber glass (or clear glass covered with aluminum foil) and stopper linings are made of aluminum (aluminum foil between sample and stopper) or Teflon®

Table E.2
Suggested volumes and preservation methods for bioassays of sediment samples

End use	Type of container	Wet weight or sample volume	Storage conditions	
			Temperature	Maximum storage time
Sediments to be used for toxicity assays and possibly contaminated with metals	Teflon® Glass High-density polyethylene bags or containers	1–3 L*	4 ± 2°C	≤ 6 weeks preferably ≤ 2 weeks
Sediments to be used for toxicity assays and possibly contaminated with organic matter	Glass with polyethylene- or aluminum-lined stopper Teflon® Stainless steel High-density polyethylene bags or containers	1–3 L*	4 ± 2°C	≤ 6 weeks preferably ≤ 2 weeks
Control and reference sediments for toxicity assays	Teflon® Glass High-density polyethylene bags or containers	> 15 L	4 ± 2°C	≤ 12 months**

(Modified from Environment Canada, 1994)

* Depending on type and number of bioassays

** These sediments must be monitored during this period to ensure they are acceptable despite any possible changes in their physicochemical characteristics.

Table E.3
Recommended pre-treatment for sediment sampling equipment and containers

Inorganic contaminants

- Scrub with phosphate-free soap and hot water.
- Rinse with high-pressure hot-water jet.
- Soak for 72 hours in an acid bath of HNO_3 8 M (50 mL of HNO_3 per litre).
- Rinse four times with hot water.
- Rinse three times with distilled deionized water.
- Wash Teflon® stoppers (or Teflon®-lined stoppers) with soap and hot water and then rinse with distilled deionized water.

Organic contaminants

- Scrub with phosphate-free soap and hot water.
- Rinse with high-pressure hot-water jet.
- If necessary, soak for 72 hours in an acid bath of HNO_3 8 M (50 mL of HNO_3 per litre).
- Rinse four times in hot tap water.
- Rinse three times in distilled deionized water.
- Rinse twice in acetone* (purity appropriate to pesticide analysis).
- Rinse twice in hexane.
- Evaporate solvents under a fume hood.
- Rinse aluminum foil (or Teflon® lining) twice in petroleum ether, then let dry under fume hood.
- Wash bottles with soap and hot water and then rinse with distilled deionized water.
- Wash bottles with soap and hot water and then rinse with distilled deionized water.
- Wash bottles with soap and hot water and then rinse with distilled deionized water.
- Cut out aluminum (or Teflon®) liners with scissors washed in acetone.
- Cut out aluminum (or Teflon®) liners with scissors washed in acetone.
- Cut out aluminum (or Teflon®) liners with scissors washed in acetone.
- Insert clean aluminum foil (or Teflon® liner) between stopper and bottle.

(Environment Canada, 1994)

* Heat-resistant bottles (to 350°C) can also be put in an oven to evaporate organic solvent residues.

Table E.4
Recommended container types and conditions for storing pore water samples

End use	Type of container	Wet weight or sample volume	Storage conditions	
			Temperature	Maximum storage time
Main ions and elements in pore water Ca, Mg, Cl, Si, Fl, Na, SO_4, K, Al, Fe, acidity, alkalinity	Teflon®	40 mL	-20°C	≤ 6 weeks
	Amber glass with Teflon®-lined stopper			
	High-density polyethylene containers			
Nutrients in pore water: Ammonia nitrogen, nitrous nitrogen, nitric nitrogen, total organic carbon, soluble reactive phosphorus, dissolved inorganic carbon, dissolved organic carbon	Amber glass with Teflon®-lined stopper	40 mL	-20°C	≤ 6 months
P (total)	Amber glass with Teflon®-lined stopper	40 mL	-20°C or 4 ± 2°C with 1 mL of H_2SO_4 at 30% per 100 mL	≤ 6 weeks ≤ 2 weeks
Trace elements (total) in pore water: Ba, Be, Cd, Cr, Cu, Co, Li, Mn, Mo, Ni, Pb, Sb, Sr, Va, Zn	Teflon® Polyethylene	10–250 mL	-20°C or 4 ± 2°C with 2 mL of HNO_3 1 M per 1000 mL of pore water	≤ 6 months ≤ 6 weeks
Ag	Amber polyethylene	250 mL	4 ± 2°C with 1 g of Na_2-EDTA per 250 mL of pore water	≤ 6 weeks
Hg	Teflon® Glass (Soviral/ Wheaton)	100 mL	4 ± 2°C with 1 mL of H_2SO_4 per 100 mL of pore water	≤ 6 weeks
Organic contaminants in pore water[a]	Amber glass with aluminum-lined stopper Amber glass with Teflon®-lined stopper	1000 mL[b]	4 ± 2°C, acidified with H_2SO_4 or 10 g of Na_2SO_4 added per litre of pore water	14 days

Table E.4 (cont'd.)
Recommended container types and conditions for storing pore water samples

Parameter	Type of container	Wet weight or sample volume	Storage conditions	
			Temperature	Maximum storage time
Organochlorines and PCBs	Teflon®	1000 mL	-20°C or 4 ± 2°C	≤ 6 month
	Amber glass with Teflon®-lined stopper			≤ 6 weeks
	High density polyethylene containers			
Organophosphorus compounds	Amber glass with Teflon®-lined stopper	1000 mL	-20°C or 4 ± 2°C, acidified with HCl to pH 4.4	≤ 6 months ≤ 6 weeks
PCP	Amber glass with Teflon®-lined stopper	1000 mL	-20°C or 4 ± 2°C, acidified with H_2SO_4 to pH < 4 or preserved with 0.5 g of $CuSO_4$ per litre of pore water	≤ 6 months ≤ 6 weeks
Phenoxy herbicides	Teflon®	1000 mL	-20°C or 4 ± 2°C, acidified to pH < 2 with H_2SO_4	≤ 6 months
	Polyethylene			≤ 6 weeks
Polyaromatic hydrocarbons	Amber polyethylene	1000 mL	-20°C or 4 ± 2°C	≤ 6 months ≤ 6 weeks
Pore water[c] or elutriate for toxicity assays	Teflon®	1–3 L[d]	4 ± 2°C	≤ 72 hours
	Glass (Soviral/ Wheaton)			
	Amber glass with aluminum-lined stopper			
	Amber glass with Teflon®-lined stopper			

(Modified from Environment Canada, 1994)

a It is very difficult to collect enough pore water for analyses of volatile and aromatic organic compounds.
b 2–15 L of sediments
c It is very difficult to collect enough pore water for standard toxicity bioassays; however, smaller quantities will suffice if the study design allows for the extraction of successive sediment samples or use of composite samples obtained from repeated sampling of the same station. It should be remembered that if pore water collected on site is exposed to oxygen (i.e. air), its geochemical properties will be modified (Mudroch 1992).
d 2–15 L of sediments.

Table E.5
Recommended pre-treatment for pore-water sampling equipment and containers

Inorganic contaminants and trace metals

- Scrub stoppers and bottles with a brush, phosphate-free soap and hot water.
- Rinse twice with hot water.
- Rinse three times with distilled deionized water.
- Soak for 72 hours in acid bath of HNO_3 8 M (50 mL of HNO_3 per litre).
- Rinse three times in distilled deionized water.

Nonvolatile organic contaminants

- Scrub with phosphate-free soap and hot water.
- Rinse with high-pressure hot-water jet.
- Rinse three times with distilled deionized water.
- Rinse twice in acetone* (purity appropriate to pesticide analysis).
- Evaporate solvents under fume hood.
- Rinse aluminum foil (or Teflon® lining) twice in petroleum ether and allow to dry under fume hood.
- Wash bottles with soap and hot water and rinse with distilled deionized water.
- Cut out aluminum (or Teflon®) liners with scissors washed in acetone.
- Insert clean sheet of aluminum foil (or Teflon® liner) between stopper and bottle.

Volatile organic contaminants

- Scrub with phosphate-free soap and hot water.
- Rinse with high-pressure hot-water jet.
- Rinse three times with distilled deionized water.
- Rinse twice in acetone** (purity appropriate to pesticide analysis).
- Rinse twice in hexane (purity appropriate to pesticide analysis).
- Evaporate solvents under fume hood.***
- Wash Teflon®-lined cap septa in soapy water.
- Rinse thoroughly in hot water.
- Rinse four or five times in distilled deionized water.
- Replace cleaned cap septa after each use.
- Replace cleaned cap septa after each use.
- Ensure that side lined with Teflon® (white) of each cap septa is on the bottom.

(Environment Canada, 1994)

* Heat-resistant bottles and stoppers (to 350°C) can also be put in an oven to evaporate organic solvent residues.

** It is very difficult to collect enough pore water for standard toxicity bioassays; however, smaller quantities will suffice if the study design allows the extraction of successive sediment samples or use of composite samples obtained from the repeated sampling of the same station. It should be remembered that if pore water collected on site is exposed to oxygen (i.e. air), its geochemical properties will be modified (Mudroch 1992).

*** Sauger et al. (1990) developed a method in which the filter is integrated with the centrifuge tube, so no glove box is required. This allows 75% of the water to be recovered from sandy sediments, compared with only 25–30% with squeezing.